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METHODS FOR TESTING PETROLEUM PRODUCTS

Adopted by the
Interdepartmental Petroleum
Specifications Committee

A HANDBOOK FOR INSPECTING LABORATORIES

WASHINGTON
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METHODS FOR TESTING PETROLEUM PRODUCTS.

Adopted by the INTERDEPARTMENTAL PETROLEUM SPECIFICATIONS COMMITTEE.

INTRODUCTION.

The methods included in this handbook are used officially in the routine testing and inspection of petroleum products bought under Federal specifications. They supersede similar methods published in Bulletins 1 to 5, inclusive, of the Committee on Standardization of Petroleum Specifications.

The Interdepartmental Petroleum Specifications Committee was formed under authority of Circular 42 of the Bureau of the Budget, dated October 10, 1921, and Executive Order 3578, dated November 8, 1921. It replaced the Interdepartmental Committee on Standardization of Petroleum Specifications, which in turn superseded the war-time Committee on Standardization of Petroleum Specifications. The work of the several committees has been continuous, and the present set of testing methods is the result of several years' experience in preparing specifications and drawing up testing methods for petroleum products.

The present set of testing methods was prepared by the Technical Committee on Standardization of Petroleum Specifications and adopted by the Interdepartmental Committee on Standardization of Petroleum Specifications, which were the immediate predecessors of the present committee. On October 10, 1921, when they ceased to function, the personnel of the two committees was as follows:

Dept. or board.	Interdepartmental committee	Technical committee.
Interior	Dr. H. Foster Bain, chairman	Mr. N. A. C. Smith, chairman.
Agriculture	Mr. B. A. Anderton	Mr. B. A. Anderton.
Shipping Board	Mr. M. W. Bowen	Mr. G. M. Talbot.
Post Office	Mr. E. B. Cranford	Mr. E. B. Cranford.
Panama Canal	Mr. W. A. E. Doying	Mr. W. A. E Doying.
War	Capt. Wm. H. Lee	Capt. Wm. H. Lee.
Navy	Commander H. A. Stuart	Commander H. A. Stuart.
Treasury	Mr. J. H. Vawter	Mr. J. H. Vawter.
Commerce	Dr. C. W. Waidner	Dr. H. C. Dickinson.

These committees felt that they should maintain close cooperation with the various industries interested in the production and use of petroleum products. Accordingly, the following advisory board was constituted, consisting of representatives of several engineering societies:

Society.	Representative.
American Society for Testing Materials	Dr. T. G. Delbridge, chairman.
Independent Oil Men's Association	Mr. E. E. Grant.
Society of Automotive Engineers	Mr. G. A. Green.
Western Petroleum Refiners' Association	Mr. H. G. James.
National Petroleum Association	Mr. O. P. Keeney.
American Petroleum Institute	Dr. Van. H. Manning.
American Society of Mechanical Engineers	Mr. H. A. S. Howarth.

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The present committee, in adopting the testing methods, gratefully acknowledges its indebtedness to the previous committees and to the advisory board. The cooperation of the American Society for Testing Materials, a number of whose methods have been adopted, is also acknowledged.

INTERDEPARTMENTAL PETROLEUM SPECIFICATIONS COMMITTEE.

Department or board.	Committee members.
Interior	Mr. N. A. C. Smith, chairman.
Agriculture	Mr. B. A. Anderton.
Shipping Board	Mr. G. M. Talbot.
Post Office	Mr. E. B. Cranford.
Panama Canal	Mr. W. A. E. Doying.
War	Capt. Wm. H. Lee.
Navy	Commander H. A. Stuart.
Treasury	Mr. J. H. Vawter.
Commerce	Mr. W. S. James.

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METHOD 10.1.—COLOR BY SAYBOLT CHROMOMETER.

APPARATUS.

Saybolt chromometer (see fig. 1), consisting of two similar glass tubes 20 inches long and about $\frac{5}{8}$ inch in internal diameter. One tube is open at both ends, the other (the oil tube) is permanently closed at the bottom with a colorless glass disk, and is provided with a pet cock on one side at the bottom. The tubes are supported in a vertical position above a mirror arranged to reflect light upward through the tubes. Above the tubes is an eyepiece so designed that the field of vision is equally divided between the two tubes. A standard yellow glass disk is placed at the bottom of the open tube.

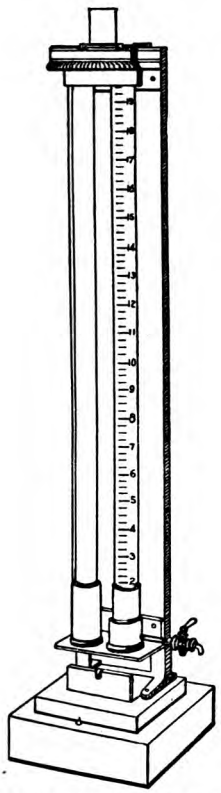

FIGURE 1.—Saybolt universal chromometer.

PROCEDURE.

Place the apparatus at a north window so that only direct light from the sky is reflected upward through the tubes from the mirror. Clean the oil tube by running through it some of the sample to be tested. Fill the oil tube with the sample to be tested and compare the color of the 20-inch column of liquid with that of the standard yellow disk. If it is lighter than the standard, report it as +25 Saybolt. If it is equal to the standard, report it as 25 Saybolt. If it is darker than the standard, draw off 2 inches of liquid and compare the color of the 18-inch column with the standard. If equal to standard, report as 24 Saybolt. If darker than standard, again draw off liquid in accordance with the following table:

Saybolt universal chromometer table for determination of color shades of refined oil.

	Oil in tube.	Color shade.		Oil in tube.	Color shade.
	Inches.	*Number.*		*Inches.*	*Number.*
One disk	20	25	One disk	$10\frac{6}{8}$	20
Do	18	24	Do	$9\frac{5}{8}$	19
Do	16	23	Do	$8\frac{3}{4}$	18
Do	14	22	Do	$7\frac{7}{8}$	17
Do	12	[a] 21	Do	$6\frac{7}{8}$	[b] 16

[a] No. 21 color on the Saybolt chromometer is equal to the color of an aqueous solution of potassium bichromate containing 0.0048 gram per liter.

[b] No. 16 color on the Saybolt chromometer is equal to the color of an aqueous solution of potassium bichromate containing 0.0120 gram per liter.

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As a check, draw off liquid in accordance with the table to lighten the color of the column one shade after it matches the standard.

By using two disks and a table furnished by the manufacturer the instrument may be used to measure the color of darker oils. Keep the instrument covered when not in use.

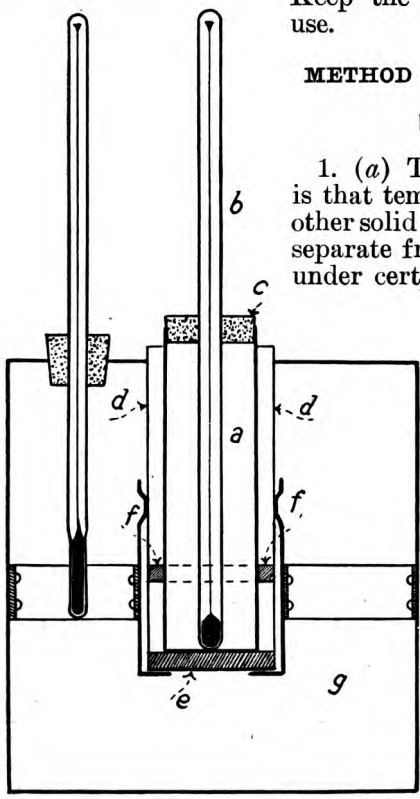

FIGURE 2.—Apparatus for cloud and pour test: *a*, Test jar; *b*, thermometer; *c*, cork; *d*, jacket; *e*, disk of cork or felt; *f*, ring gasket; *g*, cooling bath.

METHOD 20.1.—CLOUD AND POUR POINTS.

[A. S. T. M. Method D97–21T.]

1. (*a*) The cloud point of a petroleum oil is that temperature at which paraffin wax or other solid substances begin to crystallize out or separate from solution when the oil is chilled under certain definite specified conditions.

(*b*) The pour point of a petroleum oil is the lowest temperature at which this oil will pour or flow when it is chilled without disturbance under certain definite specified conditions.

2. (*a*) The test for cloud point shall be used only for oils which are transparent in layers 1½ inches thick.

(*b*) The test for pour point shall be used for all other petroleum oils and may be used for oils on which the test for cloud point is permitted.

APPARATUS.

3. The test jar *a* shall be of clear glass, cylindrical form, flat bottom, approximately 1¼ inches in inside diameter and 4½ to 5 inches high. An ordinary 4-ounce oil sample bottle may be used if the test jar is not available.

4. The thermometer *b* shall conform to the following specifications:

Type: Etched stem glass.
Total length: 222 mm.
Stem: Plain front, enamel back, suitable thermometer tubing; diameter, 7 to 8 mm.
Bulb: Corning normal, Jena 16 III, or equally suitable thermometric glass; maximum length, 9.5 mm.; diameter, not greater than stem.
Actuating liquid: Mercury.
Range: −36° to +120° F.
Immersion: 4¼ inches; the words "4¼ in. Immersion" shall be etched on tube, and a line etched around the stem to indicate the depth of immersion.
Distance to −36° line from bottom of bulb: 120 to 130 mm.
Distance to 120° line from top of stem: 19 to 25 mm.
Expansion chamber: To hold 212° F.
Filled: Nitrogen gas.
Top finish: Plain.

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Graduation: All lines, figures, and letters clear cut and distinct; scale graduated in 2° F. divisions; scale numbered every 20° F., starting at —20°. The —30° line and each succeeding 10° line to be longer than the others.

Special markings: "A. S. T. M. cloud and pour," Serial Number, and manufacturer's name or trade mark etched on the stem.

Accuracy: Error at any point on scale shall not exceed one smallest scale division.

Points to be tested for certification: —28°, +32°, +92° F.

5. The cork, *c*, shall fit the test jar, and shall be bored centrally to take the test thermometer.

6. The jacket, *d*, shall be of glass or metal and shall be water tight, of cylindrical form, flat bottom, about 4½ inches deep, with inside diameter ½ inch greater than outside diameter of the test jar.

7. A disk of cork or felt, *e*, ¼ inch thick and of the same diameter as the inside of the jacket will be required.

8. The ring gasket, *f*, shall be about $\frac{3}{16}$ inch thick, and made to fit snugly around the outside of the test jar and loosely inside the jacket. This gasket may be made of cork, felt, or other suitable material, elastic enough to cling to the test jar and hard enough to hold its shape. The purpose of the ring gasket is to prevent the test jar from touching the jacket.

9. The cooling bath, *g*, shall be of a type suitable for obtaining the required temperatures. The size and shape of the bath are optional but a support, suitable for holding the jacket firmly in a vertical position, is essential. The required bath temperatures may be maintained by refrigeration if available, otherwise by suitable freezing mixtures.

NOTE.—The freezing mixtures commonly used are as follows:
For temperatures down to 35° F., ice and water.
For temperatures down to —5° F., crushed ice and sodium chloride.
For temperatures down to —25° F., crushed ice and calcium chloride.
For temperatures down to —70° F., solid carbon dioxide and acetone.
The last named mixture may be made as follows: In a covered metal beaker chill a suitable amount of acetone to 10° F., or lower, by means of an ice-salt mixture. Invert a cylinder of liquid carbon dioxide and draw off carefully into a chamois-skin bag the desired amount of carbon dioxide, which through rapid evaporation will quickly become solid. Then add to the chilled acetone enough of the solid carbon dioxide to give the desired temperature.

PROCEDURE FOR CLOUD POINT.

10. The oil to be tested shall be brought to a temperature at least 25° F. above the approximate cloud point. Moisture, if present, shall be removed by any suitable method, as by filtration through dry filter paper until the oil is perfectly clear, but such filtration shall be made at a temperature at least 25° F. above the approximate cloud point.

The clear oil shall be poured into the test jar, *a*, to a height of not less than 1 nor more than 1¼ inches. The test jar may be marked to indicate the proper level.

The test jar shall be tightly closed by the cork, *c*, carrying the test thermometer, *b*, in a vertical position in the center of the jar with the thermometer bulb resting on the bottom of the jar.

The disk, *e*, shall be placed in the bottom of the jacket, *d*, and the test jar with the ring gasket, *f*, 1 inch above the bottom shall be inserted into the jacket. The disk, jacket, and inside of the jacket shall be clean and dry.

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The temperature of the cooling bath, *g*, shall be adjusted so that it is below the cloud point of the oil by not less than 15° nor more than 30° F. and this temperature shall be maintained throughout the test. The jacket, containing the test jar, shall be supported firmly in a vertical position in the cooling bath so that not more than 1 inch of the jacket projects out of the cooling medium.

At each test thermometer reading which is a multiple of 2° F. the test jar shall be removed from the jacket quickly but without disturbing the oil, inspected for cloud, and replaced in the jacket. This complete operation shall require not more than three seconds.

When the bottom of the oil has become opaque to a height of not less than $\frac{1}{8}$ nor more than $\frac{3}{16}$ inch, the reading of the test thermometer, corrected for error if necessary, shall be recorded as the cloud point. The required height of cloud is approximately at the middle of the thermometer bulb. The test jar may be marked to indicate the proper level.

PROCEDURE FOR POUR POINT.

11. Oils having a viscosity greater than 600 seconds, Saybolt universal at 100° F., shall be allowed to stand in the test jar at a temperature of 60° to 85° F. for at least five hours prior to making the test for pour point. A viscous oil which has been stored in a warm place is liable to show an abnormally low fictitious pour point unless this precaution is observed. Oils having a viscosity not greater than 600 seconds, Saybolt universal at 100° F., may be tested without such preliminary standing.

After preliminary standing, if necessary, the oil to be tested shall be brought to a temperature of 90° F., or to a temperature 15° F. higher than its pour point, if this pour point is above 75° F., and shall be poured into the test jar, *a*, to a height of not less than 2 nor more than $2\frac{1}{4}$ inches. The jar may be marked to indicate the proper level.

The test jar shall be tightly closed by the cork, *c*, carrying the test thermometer, *b*, in a vertical position in the center of the jar with the thermometer bulb immersed so that the beginning of the capillary shall be $\frac{1}{8}$ inch below the surface of the oil.

The disk, *e*, shall be placed in the bottom of the jacket, *d*, and the test jar, with the ring gasket, *f*, 1 inch above the bottom, shall be inserted into the jacket. The disk, gasket, and inside of jacket shall be clean and dry.

The temperature of the cooling bath, *g*, shall be adjusted so that it is below the pour point of the oil by not less than 15 nor more than 30° F., and this temperature shall be maintained throughout the test. The jacket containing the test jar shall be supported firmly in a vertical position in the cooling bath so that not more than 1 inch of the jacket projects out of the cooling medium.

At each test thermometer reading, which is a multiple of 5° F., the test jar shall be removed from the jacket carefully and shall be tilted just sufficiently to ascertain whether the oil around the thermometer remains liquid. As long as the oil around the thermometer flows when the jar is tilted slightly the test jar shall be replaced in the jacket. The complete operation of removal and replacement shall

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require not more than three seconds. As soon as the oil around the thermometer does not flow when the jar is tilted slightly, the test jar shall be held in a horizontal position for exactly five seconds and observed carefully. If the oil around the thermometer shows any movement under these conditions, the test jar shall be immediately replaced in the jacket and the same procedure shall be repeated at the next temperature reading 5° F. lower. As soon as a temperature is reached at which the oil around the thermometer shows no movement for exactly five seconds the test shall be stopped.

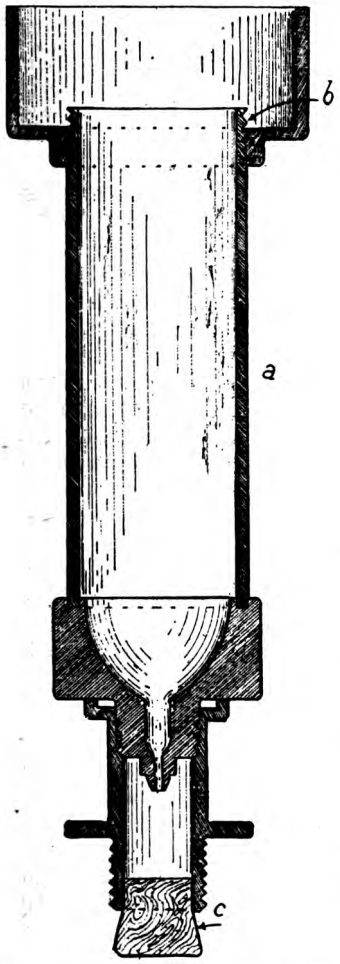

FIGURE 3.—Sectional view of standard oil tube, Saybolt standard universal viscosimeter: *a*, Tube; b, overflow cup; *c*, cork.

The lowest reading of the test thermometer, corrected for error if necessary, at which the oil around the thermometer shows any movement when the test jar is held in a horizontal position for exactly five seconds shall be recorded as the pour point.

METHOD 30.1.—VISCOSITY OF LUBRICATING OILS.

[A. S. T. M. Method D47–21.]

APPARATUS.

1. Viscosity shall be determined by means of the Saybolt standard universal viscosimeter.

2. (*a*) The Saybolt standard universal viscosimeter (see fig. 3) is made entirely of metal. The standard oil tube is fitted at the top with an overflow cup, *b*, and the tube is surrounded by a bath. At the bottom of the standard oil tube is a small outlet tube through which the oil to be tested flows into a receiving flask (see fig. 4), whose capacity to a mark on its neck is 60 (±0.15) c. c. The lower end of the outlet tube is inclosed by a large V tube, which when stoppered by a cork, *c*, acts as a closed air chamber and prevents the flow of oil through the outlet tube until the cork is removed and the test started. A looped string may be attached to the lower end of the cork as an aid to its rapid removal. The temperatures in the standard oil tube and in the bath are shown by thermometers. The bath may be heated by any suitable means. The standard oil tube shall be thoroughly cleaned, and all oil entering the standard oil tube shall be strained through a 60-mesh wire strainer. A stop watch shall be used for taking the time of flow of the oil and a pipette shall be used for draining the overflow cup of the standard oil tube.

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(*b*) The standard oil tube, which may be standardized by the U. S. Bureau of Standards, Washington, D. C., shall conform to the dimensions given below:

Dimensions of oil tube for viscosimeter.

Description.	Minimum.	Normal.	Maximum.
	Cm.	*Cm.*	*Cm.*
Inside diameter of outlet tube	0.1750	0.1765	0.1780
Length of outlet tube	1.215	1.225	1.235
Height of overflow rim above bottom of outlet tube	12.40	12.50	12.60
Diameter of container of standard oil tube	2.955	2.975	2.995
Outer diameter of outlet tube at lower end	.28	.30	.32

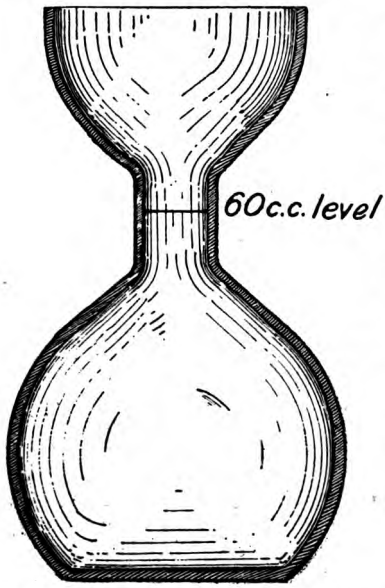

FIGURE 4.—Sectional view of receiving flask for use with Saybolt standard universal and Saybolt Furol viscosimeters (A. S. T. M.).

3. Viscosity shall be determined at 100° F. (37.8° C.), 130° F. (54.4° C.), or 210° F. (98.9° C.). The bath shall be held constant within 0.25° F. (0.14° C.) at such a temperature as will maintain the desired temperature in the standard oil tube. For viscosity determinations at 100° and 130° F., oil or water may be used as the bath liquid. For viscosity determinations at 210° F., oil shall be used as the bath liquid. Viscosity determinations shall be made in a room free from drafts and from rapid changes in temperature. All oil introduced into the standard oil tube, either for cleaning or for test, shall first be passed through the strainer.

PROCEDURE.

To make the test, heat the oil to the necessary temperature, and clean out the standard oil tube. Pour some of the oil to be tested through the cleaned tube. Insert the cork stopper into the lower end of the air chamber at the bottom of the standard oil tube sufficiently to prevent the escape of air, but not to touch the small outlet tube of the standard oil tube.

Heat the oil to be tested, outside the viscosimeter, to slightly below the temperature at which the viscosity is to be determined and pour it into the standard oil tube until it ceases to overflow into the overflow cup. By means of the oil-tube thermometer keep the oil in the standard oil tube well stirred and also stir well the oil in the bath. It is extremely important that the temperature of the bath be maintained constant during the entire time consumed in making the test. When the temperature of the bath and of the oil in the standard oil tube are constant and the oil in the standard oil tube is at the desired temperature, withdraw the oil-tube thermometer; quickly remove the surplus oil from the overflow cup by means of a pipette so that the

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level of the oil in the overflow cup is below the level of the oil in the tube proper; and place the 60-c. c. flask in position so that the stream of oil from the outlet tube will strike the neck of the flask so as to avoid foam. Snap the cork from its position, and at the same instant start the stop watch. Stir the liquid in the bath during the run and carefully maintain it at the previously determined proper temperature. Stop the watch when the bottom of the meniscus of the oil reaches the mark on the neck of the receiving flask.

The time in seconds for the delivery of 60 c. c. of the oil is the Saybolt viscosity of the oil at the temperature at which the test was made.

METHOD 30.2.—VISCOSITY OF FUEL OILS AT 122° F.

[A. S. T. M. Method D88–21T.]

APPARATUS AND PROCEDURE.

1. Viscosity shall be determined by the Saybolt Furol viscosimeter.

2. The apparatus and method of operation shall be the same as for the standard Saybolt universal viscosimeter (see method 30.1) as given in section 2 of the standard test for viscosity of lubricants (serial designation D47) of the American Society for Testing Materials, all dimensions being the same except the diameter of the outlet tube, which shall be as follows:

	Minimum.	Normal.	Maximum.
Inside diameter of outlet tube.....centimeters..	0.313	0.315	0.317
Outside diameter at lower end.....do....	.40	.43	.46

3. Viscosity shall be determined at 122° F. (50° C.) and shall be expressed in seconds, Saybolt Furol at 122° being the time in seconds for the delivery of 60 c. c. of oil.

4. Oil showing a time of less than 25 seconds, Saybolt Furol, at 122° F., shall be tested on the Saybolt universal at 122° F. Oil showing a time of less than 32 seconds Saybolt universal, at 122° F., shall be measured in the Saybolt universal at 100° F. (37.8° C.). This test does not apply to fuels having a viscosity at 100° F. of less than 32 seconds, Saybolt universal, which are not considered to be fuel oils.

METHOD 30.3.—VISCOSITY OF FUEL OILS AT 77° F.

APPARATUS AND PROCEDURE.

The apparatus and procedure are the same as in method 30.2 except that paragraph 3 shall read as follows:

3. Viscosity shall be determined at 77° F. and shall be expressed in seconds, Saybolt Furol at 77° F. being the time in seconds for the delivery of 60 c. c. of oil.

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METHOD 40.1.—MELTING OF PARAFFIN WAX.

[A. S. T. M. Method D87–21T.]

DEFINITION.

1. The A. S. T. M. Paraffin-Wax Melting Point is the temperature at which melted paraffin wax, when allowed to cool under definite specified conditions, first shows a minimum rate of temperature change.

APPARATUS.

2. *Wax container.*—Test tube of standard form, 25 mm. (1 inch) outside diameter and 100 mm. (4 inches) long. It may be marked with a filling line 2 inches above the bottom. This test tube shall be closed by a tightly fitting cork having two openings, one at the center for the melting-point thermometer and the other for a stirrer at one side of the center. The opening for the stirrer may be lined with glass or metal tubing to act as a guide for the stirrer.

3. *Air bath.*—Suitable water-tight cylinder, 2 inches in inside diameter and 4½ inches deep. This air bath shall be provided with a tightly fitting cork having a central opening for holding the test tube firmly in a vertical position in the center of the air bath.

4. *Water bath.*—Suitable cylinder, 5⅛ inches in inside diameter and 6 inches deep. This water bath shall be provided with a suitable cover and with the guides and fasteners necessary to hold the air bath firmly in a vertical central position so that the sides and bottom of the air bath shall be surrounded by a layer of water 1½ inches thick. The water-bath cover shall have a slot for introduction of a suitable stirrer and shall have an opening for the bath thermometer so that the latter may be suspended in a vertical position ¾ inch from the outside wall of the water bath. Air bath, water bath, and water-bath cover may be conveniently made of metal in one assembly, as shown in figure 5.

5. *Stirrer in test tube.*—Brass or copper wire, ⅛ inch in diameter and about 12 inches long. A circular loop, ½ inch in diameter, shall be formed at one end of this wire in such a manner that the loop lies in a horizontal plane when the rest of the wire is in a vertical position. The stirrer thus formed shall be passed through the proper opening in the test-tube cork and the upper end may then be bent into a shape convenient for holding.

6. *Thermometer.*—The A. S. T. M. paraffin-wax melting-point thermometer shall conform to the following specifications:

Type: Etched stem glass.
Total length: 368 mm.
Stem: Plain front, enamel back, suitable thermometer tubing; diameter 6 to 7 mm.
Bulb: Corning normal, Jena 16 III, or equally suitable thermometric glass; length, maximum, 28 mm.; diameter, not greater than stem.
Actuating liquid: Mercury.
Range: 80° to 160° F.
Immersion: 3⅛ inches; the words "3⅛ in. Immersion" shall be etched on the stem and also a line around the stem to indicate the depth of immersion.
Distance to 80° line from bottom of bulb: 105 to 115 mm.
Distance to 160° line from top of stem: 25 to 40 mm.
Contraction chamber: Top to be not more than 41 mm. from bottom of bulb.

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Expansion chamber: To hold 212° F.
Filled: Nitrogen gas.
Top finish: Plain.
Graduation: All lines, figures, and letters to be clean cut and distinct; scale graduation in 0.2° F. and numbered every 2° F., every full degree line to be longer than the others.
Special markings: "A. S. T. M. Pffe. M. P.," Serial Number, and manufacturer's trade mark or name etched on the stem.
Accuracy: Error at any point on the scale shall not exceed one smallest scale division.
Points to be tested for certification: 80°, 100°, 120°, 140°, 160° F.

7. Bath thermometer of any suitable type, accurate to 2° F. throughout the required range.

PROCEDURE.

8. An average sample of the wax to be tested shall be melted in a suitable container in a water bath whose temperature shall be not more than 35° F. above the approximate melting point of the wax sample. Direct heat, such as a flame or hot plate, shall not be used and the wax sample shall not be held in the melted condition any longer than necessary.

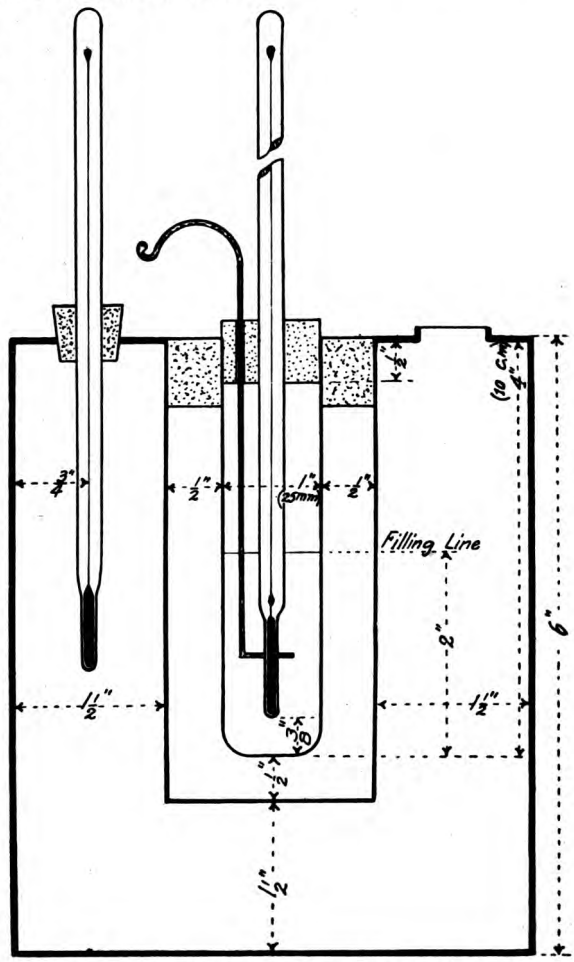

FIGURE 5.—Apparatus for determining melting point of paraffin wax.

The test tube shall be filled with melted wax to a height of 2 inches. The test-tube cork, carrying the stirrer and the melting-point thermometer with the $3\frac{1}{8}$-inch immersion line at the under surface of the cork, shall be inserted into the test tube for a distance of one-half inch. The lower end of the thermometer bulb shall then be three-eighths inch from the bottom of the test tube.

The air bath being in its proper position in the water bath, the latter shall be filled to within $\frac{1}{2}$ inch of the top with water at a temperature 15° to 20° F. below the approximate melting point of the wax sample.

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The test tube containing the melted wax, with wax stirrer and thermometer in place, shall be inserted into the air bath in a central vertical position so that the bottom of the test tube is $\frac{1}{2}$ inch from the bottom of the air bath. The temperature of the water bath shall be adjusted by stirring if necessary, so that it shall be lower than the temperature of the wax sample by not more than 30° F. and not less than 25° F., when the wax sample has cooled to a temperature 10° F. above its approximate melting point.

When these conditions have been obtained, temperature adjustment and stirring of the water bath shall be discontinued. The wax shall be stirred continuously during the remainder of the test, the stirring loop being moved up and down throughout the entire length of the test tube in a steady motion at the rate of 20 complete cycles per minute. The melting-point thermometer reading, estimated to 0.1° F., shall be observed and recorded every 30 seconds. The temperature of the wax will fall gradually at first, become almost constant, and then again fall gradually.

The melting-point thermometer reading, estimated to 0.1° F., shall be observed and recorded every 30 seconds for at least three minutes after the temperature again begins to fall after remaining almost constant. The record of temperature readings shall then be inspected and the average of the first four readings that lie within a range of 0.2° F. shall be considered as the uncorrected melting point. This temperature shall be corrected if necessary for error in the thermometer scale and the corrected temperature shall be reported as the "A. S. T. M. Paraffin-Wax Melting Point."

ACCURACY.

9. Duplicate determinations on the same sample should differ by not more than 0.2° F.

METHOD 40.2.—MELTING POINT OF PARAFFIN WAX.

APPARATUS.

A stand consisting of a base, at opposite ends of which are fastened parallel vertical supports. At right angles to the vertical supports are fastened three horizontal platforms with holes directly above one another. Through the holes in the top platform are suspended hangers, controllable by set screws, on which are hung four thermometers. The holes in the two lower platforms act as a guide to prevent the thermometers from moving sideways. On the base of the platform are raised rails on which is a car on wheels carrying four metal cells. Each metal cell is $7\frac{3}{4}$ inches long at the top and $6\frac{1}{2}$ inches long at the bottom, $1\frac{9}{16}$ inches wide at the top and $\frac{7}{8}$ inch wide at the bottom, and $1\frac{5}{8}$ inches deep.

Thermometer.—A thermometer approximately 12 inches long, scaled from 90° to 150° F. in 0.25° divisions, with a bulb approximately $\frac{9}{16}$ inch in diameter and $\frac{9}{16}$ inch long.

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METHOD OF TESTING.

1. Place the car *i* (fig. 6) on the track rails *h* and the cells *g* in the compartments on the car.

2. Adjust the thermometers through the holes *d* in the middle and the lower platforms, hanging them by their rings *c* on the thermometer hangers *b*, holding them in position out of and above the cells *g* by means of the hangers *b* and set screws *a*.

3. Heat the sample 10° above its melting point.

4. Pour the melted sample into the cell *g* so that it is about three-fourths full.

5. Adjust the thermometer by lowering the hanger *b* and fasten with the set screw *a*, so that the bulb *f* is well covered, but positively do not have the bulb in contact with either the sides or bottom of the metal cell *g*.

6. Move the car *i* slowly backward and forward as the mercury is falling.

7. Watch the mercury as it falls, recording the temperature every 30 seconds. Continue this operation until three consecutive readings agree. This temperature is the true melting or solidifying point of the sample under examination.

8. Raise and fasten the thermometers out of the sample with bulbs above the cells (by means of set screws *a* and thermometer hanger *b*.)

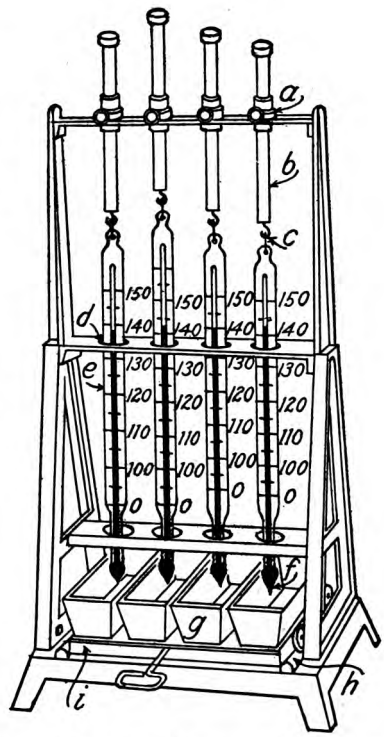

FIGURE 6.—Saybolt improved apparatus for determination of melting point: *a*, Screws; *b*, hangers; *c*, metal rings; *d*, openings for thermometers, upper and lower frames; *e*, thermometer faces; *f*, bulbs; *g*, cells; *h*, track rail; *i*, car.

METHOD 100.1.—DISTILLATION OF GASOLINE.

[A. S. T. M. Method D86–21T.]

APPARATUS.

1. *Flask.*—The standard 100-c. c. Engler flask is shown in figure 7, the dimensions and allowable tolerance being as follows:

Dimensions of Engler flask.

Description.	Centimeters.	Inches.	Tolerances (cm.).
Diameter of bulb, outside	6.5	2.56	0.2
Diameter of neck, inside	1.6	.63	.1
Length of neck	15.0	5.91	.4
Length of vapor tube	10.0	3.94	.3
Diameter of vapor tube, outside	.6	.24	.05
Diameter of vapor tube, inside	.4	.16	.05
Thickness of vapor tube wall	.1	.04	.05

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The position of the vapor tube shall be 9 cm. (3.55 inches) ±3 mm. above the surface of the liquid when the flask contains its charge of 100 c. c. The tube is approximately in the middle of the neck and set at an angle of 75° (tolerance ±3°) with the vertical.

2. *Condenser.*—The condenser (fig. 8) consists of a $\frac{9}{16}$ inch OD No. 20 Stubbs gage seamless brass tube 22 inches long. It is

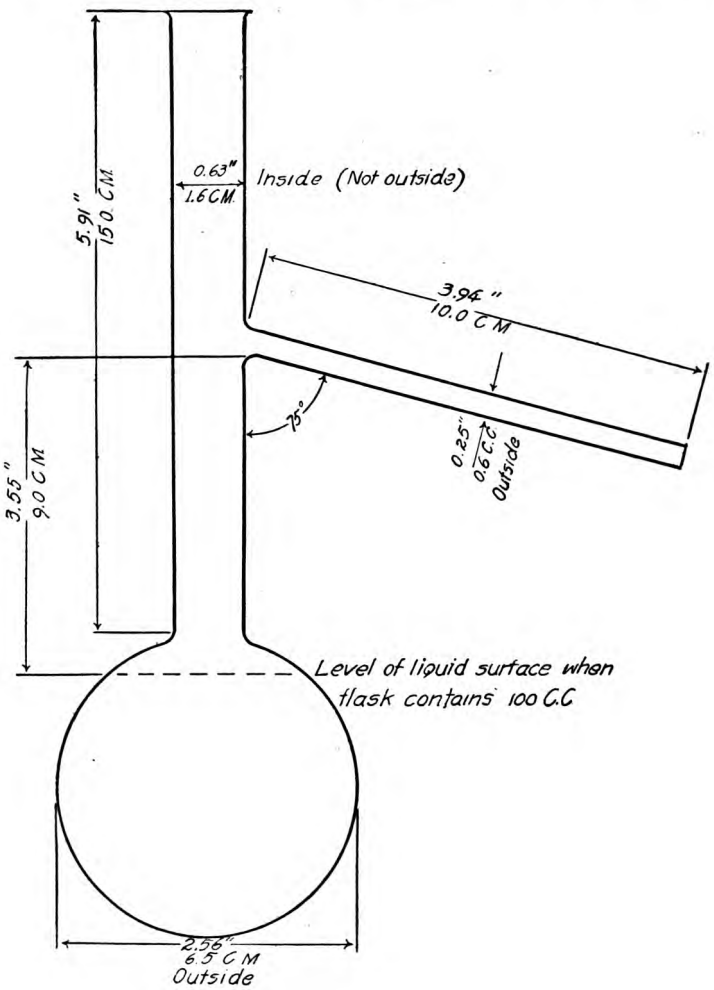

FIGURE 7.—Standard 100-c. c. Engler flask for use in making distillation tests of gasoline and kerosene.

set at an angle of 75° from the perpendicular and is surrounded with a cooling bath 15 inches long, approximately 4 inches wide by 6 inches high. The lower end of the condenser tube is cut off at an acute angle, and curved downward for a length of 3 inches and slightly backward so as to insure contact with the wall of the graduate at a point 1 to $1\frac{1}{4}$ inches below the top of the graduate when it is in position to receive the distillate.

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3. *Shield.*—The shield (fig. 8) is made of approximately 22-gage sheet metal and is 19 inches high, 11 inches long, and 8 inches wide, with a door on one narrow side, with two openings, 1 inch in diameter, equally spaced, in each of the two narrow sides, and with a slot cut in one side for the vapor tube. The centers of these four openings are 8½ inches below the top of the shield. There are also three ½-inch holes in each of the four sides, with their centers 1 inch above the base of the shield.

4. *Ring support and hard asbestos boards.*—The ring support is of the ordinary laboratory type, 4 inches or larger in diameter, and is supported on a stand inside the shield. There are two hard asbestos boards, one 6 by 6 by ¼ inch, with a hole 1¼ inches in diameter in its

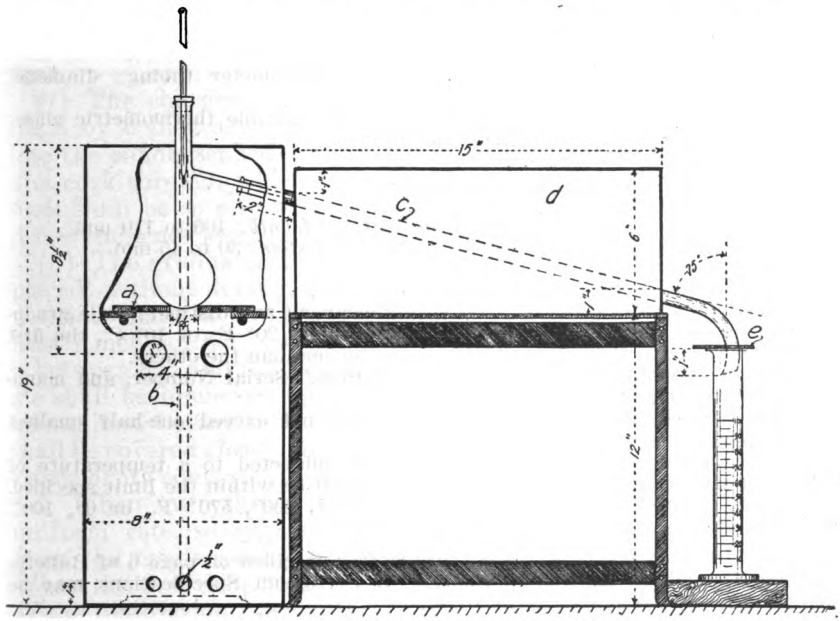

FIGURE 8.—Distillation outfit (A. S. T. M.) arranged for use of gas burner: *a*, 6 by 6 by ¼-inch asbestos board; *b*, Bunsen burner; *c*, ⅜-inch OD No. 20, Stubbs gage, brass tube; *d*, icewater bath; *e*, blotting paper.

center, the sides of which shall be perpendicular to the surface; the other, an asbestos board to fit tightly inside the shield, with an opening 4 inches in diameter concentric with the ring support. These are arranged as follows: The second asbestos board is placed on the ring and the first or smaller asbestos board on top so that it may be moved in accordance with the directions for placing the distilling flask. Direct heat is applied to the flask only through the 1¼-inch opening in the first asbestos board.

5. *Gas burner or electric heater.*—(*a*) *Gas burner.*—The burner is so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified below. The flame should never be so large that it spreads over a circle of diameter greater than 3½ inches on the under surface of the asbestos board. A sensi-

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tive regulating valve is a necessary adjunct, as it gives complete control of heating.

(*b*) *Electric heater.*—The electric heater, which may be used in place of the gas flame, shall be capable of bringing over the first drop within the time specified below when started cold, and of continuing the distillation at the uniform rate. The electric heater shall be fitted with an asbestos board, top $\frac{1}{8}$ to $\frac{1}{4}$ inch thick, having a hole $1\frac{1}{4}$ inches in diameter in the center. When an electric heater is employed the portion of the shield above the asbestos board shall be the same as with the gas burner, but the part below may be omitted.

6. *Thermometer.*—A. S. T. M. low-distillation thermometer shall conform to the following specifications:

Type: Etched stem glass.
Total length: 381 mm.
Stem: Plain front, enamel back, suitable thermometer tubing; diameter, 6 to 7 mm.
Bulb: Corning normal, Jena 16 III, or equally suitable thermometric glass; length, 10 to 15 mm.; diameter, 5 to 6 mm.
Actuating liquid: Mercury.
Range: 30° F. to 580° F., or 0° C. to 300° C.
Immersion: Total.
Distance to 30° F. or 0° C. mark, from bottom of bulb: 100 to 110 mm.
Distance to 580° F. or 300° C. mark, from top of stem: 30 to 45 mm.
Filled: Nitrogen gas.
Top finish: Glass ring.
Graduation: All lines, figures, and letters clear cut and distinct; scale graduated in 2° F., or 1° C. divisions and numbered every 20° F., or 10° C., the first and each succeeding 10° F. (5° C.) line to be longer than the others.
Special markings: "A. S. T. M. low distillation," Serial Number, and manufacturer's trade-mark etched on the stem.
Accuracy: Error at any point on scale shall not exceed one-half smallest scale division.
Test for permanency of range: After being subjected to a temperature of 560° F., or 290° C., for 24 hours the accuracy shall be within the limit specified.
Points to be tested for certification: 32°, 212°, 400°, 570° F., or 0°, 100°, 200°, 300° C.

NOTE. Until January 1, 1924, the thermometer specified on page 6 of Bulletin 5 of the Committee on Standardization of Petroleum Specifications may be used in place of the above thermometer.

7. *Graduate.*—The graduate shall be of the cylindrical type, of uniform diameter, with a pressed or molded base and a lipped top. The cylinder shall be graduated to contain 100 c. c. and the graduated portion shall be not less than 7 inches nor more than 8 inches long; it shall be graduated in single cubic centimeters, and each fifth mark shall be distinguished by a longer line. It shall be numbered from the bottom up at intervals of 10 c. c. The distance from the 100-c. c. mark to the rim shall be not less than $1\frac{1}{4}$ inches nor more than $1\frac{3}{4}$ inches. The graduations shall not be in error by more than 1 c. c. at any point on the scale.

PROCEDURE.

8. (*a*) The condenser bath shall be filled with cracked ice[1] and enough water added to cover the condenser tube. The temperature shall be maintained between 32° and 40° F. (0° and 4.45° C.).

[1] Any other convenient cooling medium may be used.

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(*b*) The condenser tube shall be swabbed to remove any liquid remaining from the previous test. A piece of soft cloth attached to a cord or copper wire may be used for this purpose.

(*c*) The bulb of the distillation thermometer shall be covered uniformly with a long-fiber absorbent cotton weighing not less than 3 nor more than 5 mg. A fresh portion of clean cotton shall be used for each distillation.

(*d*) One hundred cubic centimeters of the product shall be measured in the 100-c. c. graduated cylinder at 55° to 65° F. (12.78° to 18.33° C.) and transferred directly to the Engler flask. None of the liquid shall be permitted to flow into the vapor tube.

(*e*) The thermometer provided with a cork shall be fitted tightly into the flask so that it will be in the middle of the neck and so that the lower end of the capillary tube is on a level with the inside of the bottom of the vapor outlet tube at its junction with the neck of the flask.

(*f*) The charged flask shall be placed in the 1¼-inch opening in the 6 by 6 inch asbestos board with the vapor outlet tube inserted into the condenser tube. A tight connection may be made by means of a cork through which the vapor tube passes. The position of the flask shall be so adjusted that the vapor tube extends into the condenser tube not less than 1 inch nor more than 2 inches.

(*g*) The graduated cylinder used in measuring the charge shall be placed, without drying, at the outlet of the condenser tube in such a position that the condenser tube shall extend into the graduate at least 1 inch but not below the 100-c. c. mark. Unless the temperature is between 55° and 65° F. (12.78° and 18.33° C.) the receiving graduate shall be immersed up to the 100-c. c. mark in a transparent bath maintained between these temperatures. The top of the graduate shall be covered closely during the distillation with a piece of blotting paper or its equivalent cut so as to fit the condenser tube tightly.

9. When everything is in readiness, heat shall be applied at a uniform rate, so regulated that the first drop of condensate falls from the condenser in not less than 5 nor more than 10 minutes. When the first drop falls from the end of the condenser the reading of the distillation thermometer shall be recorded as the *initial boiling point*. The receiving cylinder shall then be moved so that the end of the condenser tube shall touch the side of the cylinder. The heat shall then be so regulated that the distillation will proceed at a uniform rate of not less than 4 nor more than 5 c. c. per minute. The reading of the distillation thermometer shall be recorded when the level of the distillate reaches each 10-c. c. mark on the graduate.

After the 90 per cent point has been recorded, the heat may be increased because of the presence of the heavy ends which have high boiling points. However, no further increase of heat should be applied after this adjustment. The 4 to 5 c. c. rate can rarely be maintained from the 90 per cent point to the end of the distillation, but in no case should the period between the 90 per cent and the end point be more than five minutes.

The heating shall be continued until the mercury reaches a maximum and starts to fall consistently. The highest temperature observed on the distillation thermometer shall be recorded as the *maximum temperature* or end point. Usually this point will be reached after the bottom of the flask has become dry.

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The total volume of the distillate collected in the receiving graduate shall be recorded as the *recovery*.

The cooled residue shall be poured from the flask into a small cylinder graduated in 0.1 c. c., measured when cool, and the volume recorded as *residue*.

The difference between 100 c. c. and the sum of the recovery and the residue shall be calculated and recorded as *distillation loss*.

ACCURACY.

10. With proper care and attention to detail, duplicate results obtained for initial boiling point and maximum temperature, respectively, should not differ from each other by more than 6° F. (3.33° C.).

METHOD 100.2.—DISTILLATION OF KEROSENE.

APPARATUS AND PROCEDURE.

The apparatus and procedure are the same as given in method 100.1, with the following exceptions:

Paragraph 4: The hard asbestos board 6 by 6 by $\frac{1}{4}$ inches shall have a hole $1\frac{1}{2}$ inches in diameter in its center.

Paragraph 6: The A. S. T. M. high-distillation thermometer shall conform to the following specifications:

Type: Etched stem glass.
Total length: 381 mm.
Stem: Plain front, enamel back, suitable thermometer tubing; diameter, 6 to 7 mm.
Bulb: Corning normal, Jena 16 III, or equally suitable thermometric glass; length, 10 to 15 mm.; diameter, 5 to 6 mm.
Actuating liquid: Mercury.
Range: 30° F. to 760° F., or 0° C. to 400° C.
Immersion: Total.
Distance to 30° F., or 0° C. mark from bottom of bulb: 25 to 35 mm.
Distance to 760° F., or 400° C. mark from top of tube: 30 to 45 mm.
Filled: Nitrogen gas.
Top finish: Glass ring.
Graduation: All lines, figures, and letters clear cut and distinct; scale graduated in 2° F. or 1° C. divisions and numbered every 20° F. or 10° C., the first and each succeeding 10° F. (5° C.) to be longer than the others.
Special markings: A. S. T. M. High Distillation, serial number, and manufacturer's name or trade mark etched on the stem.
Accuracy: Error at any point on scale shall not exceed one smallest scale division up to 700° F. or 370° C.
Tests for permanency of range: After being subjected to a temperature of 700° F. or 370° C. for 24 hours the accuracy shall be within the limit specified.
Points to be tested for certification: 32°, 212°, 400°, 700° F. or 0°, 100°, 200°, 370° C.

METHOD 110.1.—FLASH POINT OF VOLATILE FLAMMABLE LIQUIDS BY TAG CLOSED TESTER.

[A. S. T. M. Method D56–21.]

APPARATUS.

1. (*a*) The Tag closed tester shall be used for all volatile flammable liquids flashing below 175° F., with the exception of products classed as fuel oil. Determination of the flash point of fuel oil by the Tag tester is permissible, but the Pensky-Martens tester is to be preferred.

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(*b*) The Tag closed tester shall conform to the following dimensions within the limits of tolerances given:

Dimensions of Tag closed tester.

	Normal.	Tolerances.
Depth of water surface below top of cup.inches..	$1\frac{3}{32}$ (27.8 mm.).	$\pm\frac{1}{64}$ (0.4 mm.).
Depth of oil surface below top of cup.....do....	$1\frac{5}{32}$ (29.4 mm.).	$\pm\frac{1}{64}$ (0.4 mm.).
Depth of top of bulb of oil thermometer when in place below top of cup .....inches..	$1\frac{5}{16}$ (33.3 mm.).	$\pm\frac{1}{32}$ (0.8 mm.).
Inside diameter of oil cup at top.....do....	$2\frac{1}{8}$ (54.0 mm.).	±0.005 (0.1 mm.).
Weight of oil cup.....grams..	68.	±1.
Diameter of bead on top of cover.....inches..	$\frac{5}{32}$ (4.0 mm.).	$\pm\frac{1}{64}$ (0. 4 mm.).

The plane of underside of cover to be between the top and bottom of the burner tip when the latter is fully depressed.

(*c*) The thermometer shall conform to the following specifications:[2]

Type: Etched stem glass.
Total length: 275 mm.
Stem: Plain front, enamel back, suitable thermometer tubing; diameter, 6 to 7 mm.
Bulb: Corning normal, Jena 16 III, or equally suitable thermometric glass; diameter, less than stem; length, 9 to 13 mm.
Actuating liquid: Mercury.
Range: 20° F. to 230° F.
Immersion: $2\frac{1}{4}$ inches (57 mm.) from end of bulb; words "$2\frac{1}{4}$ in. Immersion" etched on the stem, also a line around the stem to indicate depth of immersion.
Distance to 20° line from bottom of bulb: 75 to 90 mm.
Distance to 230° line from top of stem: 25 to 40 mm.
Expansion chamber: Required.
Graduation: All lines, figures, and letters clear cut and distinct; scale graduated in 1° divisions; scale numbered every 10°, the first and each succeeding 5° line to be longer than the others.
Special marking: "A. S. T. M., P. M., and Tag," Serial Number, manufacturer's name or trade-mark etched on the stem.
Accuracy: Error at any point in scale shall not exceed 1° F.
Points to be tested: 32°, 100°, 150°, 212° F.

PROCEDURE.

2. (*a*) If gas is available, connect a $\frac{1}{8}$-inch rubber tube to the corrugated gas connection on the oil-cup cover. If no gas is available, unscrew the test-flame burner tip from the oil chamber on the cover and insert a wick of cotton cord in the burner tip and replace it. Put a small quantity of cotton waste in the oil chamber, insert a small quantity of signal, sperm, or lard oil in the chamber, light the wick, and adjust the flame so that it is exactly the size of the small white bead mounted on the top of the tester.

(*b*) The test shall be performed in a dim light so as to see the flash plainly.

(*c*) Surround the tester on three sides with an inclosure to keep away drafts.[3]

(*d*) See that the tester sets firm and level.

(*e*) For accuracy, the flash-point thermometers which are especially designed for the instrument should be used, as the position of the bulb of the thermometer in the oil cup is essential.

[2] This thermometer is identical with the low-range instrument specified for use with the A.S.T.M. Pensky-Martens closed tester.
[3] A shield about 18 inches square and 2 feet high, open in front, is satisfactory, but any safe precaution against all possible room drafts is acceptable. Tests made in a laboratory hood or near ventilators will give unreliable results.

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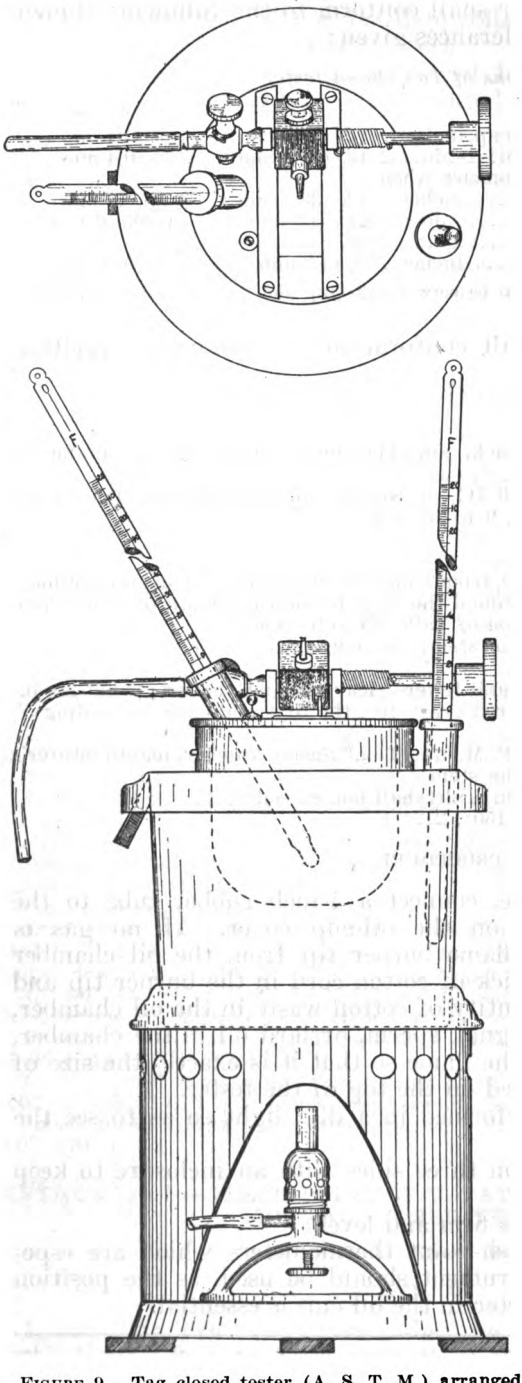

FIGURE 9.—Tag closed tester (A. S. T. M.) arranged for use of gas.

3. Put the water-bath thermometer in place and put a receptacle under the overflow spout to catch the overflow. Fill the water bath with water at such a temperature that, when testing is started, the temperature of the water bath will be at least 20° F. (11° C.) below the probable flash point of the oil to be tested.

4. Put the oil cup in place in the water bath. Measure 50 c. c. of the oil to be tested in a pipette or a graduate and place in the oil cup. The temperature of the oil shall be at least 20° F. (11° C.) below its probable flash point when testing is started. Destroy any bubbles on the surface of the oil. Put on the cover, with flash-point thermometer in place and gas tube attached. Light the pilot light on the cover and adjust the flame to the size of the small white bead on the cover.

5. Light and place the heating lamp, filled with alcohol, in the base of the tester and see that it is centrally located. Adjust the flame of the alcohol lamp so that the temperature of the oil in the cup rises at the rate of about 1.8° F. (1° C.) per minute, not faster than 2° F. (1.1° C.) nor slower than 1.6° F. (0.9 C.) per minute.

6. (*a*) Record the barometric pressure, which, in the absence of a laboratory instrument, may be obtained from the nearest Weather Bureau station.

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(*b*) Record the temperature of the oil sample at start.

(*c*) When the temperature of the oil reaches 9° F. (5° C.) below the probable flash point of the oil, turn the knob on the cover so as to introduce the test flame into the cup and turn it *promptly* back again. Do not let it snap back. *The time consumed in turning the knob down and back should be about one full second*, or the time required to pronounce distinctly the words "one-thousand-and-one."

(*d*) Record the time of making the first introduction of the test flame.

(*e*) Record the temperature of the oil sample at the time of first test.

(*f*) Repeat the application of the test flame at every 1° F. (0.5° C.) rise in temperature of the oil until there is a flash of the oil within the cup.[4]

(*g*) Record the time at which the flash point is reached.

(*h*) Record the flash point.

(*i*) If the rise in temperature of the oil from the "time of making the first introduction of the test flame" to the "time at which the flash point is reached" was faster than 2° F. (1.1° C.) or slower than 1.6° F. (0.9° C.) per minute, the test should be questioned, and the alcohol heating lamp adjusted to correct the rate of heating. It will be found that the wick of this lamp can be so accurately adjusted as to give a uniform rate of rise in temperature within the above limits and remain so.

7. (*a*) It is not necessary to turn off the test flame with the small regulating valve on the cover; leave it adjusted to give the proper size of flame.

(*b*) Having completed the preliminary test, remove the heating lamp, lift up the oil-cup cover, and wipe off the thermometer bulb. Lift out the oil cup and empty and carefully wipe it. Throw away all oil samples after they have been once used in making a test.

(*c*) Pour cold water into the water bath, allowing it to overflow into a receptacle, until the temperature of the water in the bath is lowered to 15° F. (8° C.) below the flash point of the oil, as shown by the previous test.[5]

(*d*) Place the oil cup back in the bath and measure into it a 50-c. c. charge of fresh oil. Destroy any bubbles on the surface of the oil, put on the cover with its thermometer, put in the heating lamp, record the temperature of the oil, and proceed to repeat the test as described above in sections 4 to 6, inclusive. Introduce the test flame for first time at a temperature of 10° F. (5.5° C.) below the flash point obtained on the previous test.

8. If two or more determinations agree within 1° F. (0.5° C.), the average of these results, corrected for barometric pressure, shall be considered the flash point. If two determinations do not check within 1° F. (0.5° C.), a third determination shall be made, and if the maximum variation of the three tests is not greater than 2° F. (1° C.), their average, after correcting for barometric pressure, shall be considered the flash point.

[4] Do not be misled by an enlargement of the test flame or halo around it when entered into the cup, or by slight flickering of the flame; the true flash consumes the gas in the top of the cup and causes a very slight explosion.

[5] With cold water of nearly constant temperature, it will be found that a uniform amount will be required to reduce the temperature of the water bath to the required point.

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9. Correction for barometric pressure shall only be made in cases of dispute or when the barometer reading varies more than ½ inch (13 mm.) from the standard pressure of 29.92 inches (760 mm.). When the barometer reading is below this standard pressure, add to the thermometer reading 1.6° (0.9° C.) for each inch (25 mm.) of barometer difference to obtain the true flash point. When the barometer reading is above the standard pressure, deduct 1.6° F. (0.9° C.) for each inch (25 mm.) of barometer difference to obtain the true flash point.

METHOD 110.2.—FLASH POINT BY MEANS OF THE PENSKY-MARTENS CLOSED TESTER.

[A. S. T. M. Method D93–21T.]

1. The A. S. T. M. standard Pensky-Martens closed tester shall be used for determining the flash point of fuel oil unless the use of the Tag closed tester is specified.

APPARATUS.

2. The Pensky-Martens tester, a diagram of which appears in figure 10, shall include the following major parts:

(*a*) *Cup.*—The cup of the A. S. T. M. Pensky-Martens flash tester shall be made of brass and shall satisfy the following dimensional specifications:

Specifications for cup of A. S. T. M. Pensky-Martens flash tester.

Dimensions.	Minimum.	Normal.	Maximum.	Minimum.	Normal.	Maximum.
	Inches.	*Inches.*	*Inches.*	*Cm.*	*Cm.*	*Cm.*
Inside diameter below filling mark	1.950	2.000	2.050	4.953	5.080	5.207
Difference, inside and outside diameters below filling mark	.120	.125	.130	.305	.318	.330
Inside height	2.150	2.200	2.250	5.461	5.588	5.715
Thickness of bottom	.070	.095	.120	.178	.241	.305
Distance from rim to filling mark	.845	.860	.875	2.146	2.184	2.223
Distance lower surface flange to bottom of cup	1.780	1.795	1.810	4.521	4.559	4.597

The inside of the cup may be turned to a slightly larger diameter above the filling mark and the outside may be tapered above the flange, but the wall thickness at the upper edge shall be not less than 0.04 inch (0.102 cm.). The flange should be approximately 0.5 inch (1.27 cm.) wide and approximately 0.125 inch (0.318 cm.) thick. It shall be equipped with devices for locating the position of the lid on the cup and the cup in the stove. A handle, attached permanently to the flange of the cup, is a desirable accessory.

(*b*) *Lid.*—1. *Stirring device.*—The lid shall be equipped with a stirring device consisting of a vertical steel shaft, not less than 0.1 inch (0.254 cm.) nor more than 0.125 inch (0.318 cm.) in diameter, mounted in the center of the cup, and carrying two 2-bladed brass propellers. The blades of both propellers shall be approximately 0.313 inch (0.795 cm.) wide and shall be set at an angle of approximately 45°. The smaller (upper) propeller shall have an overall diameter of approximately 0.75 inch (1.905 cm.). The larger

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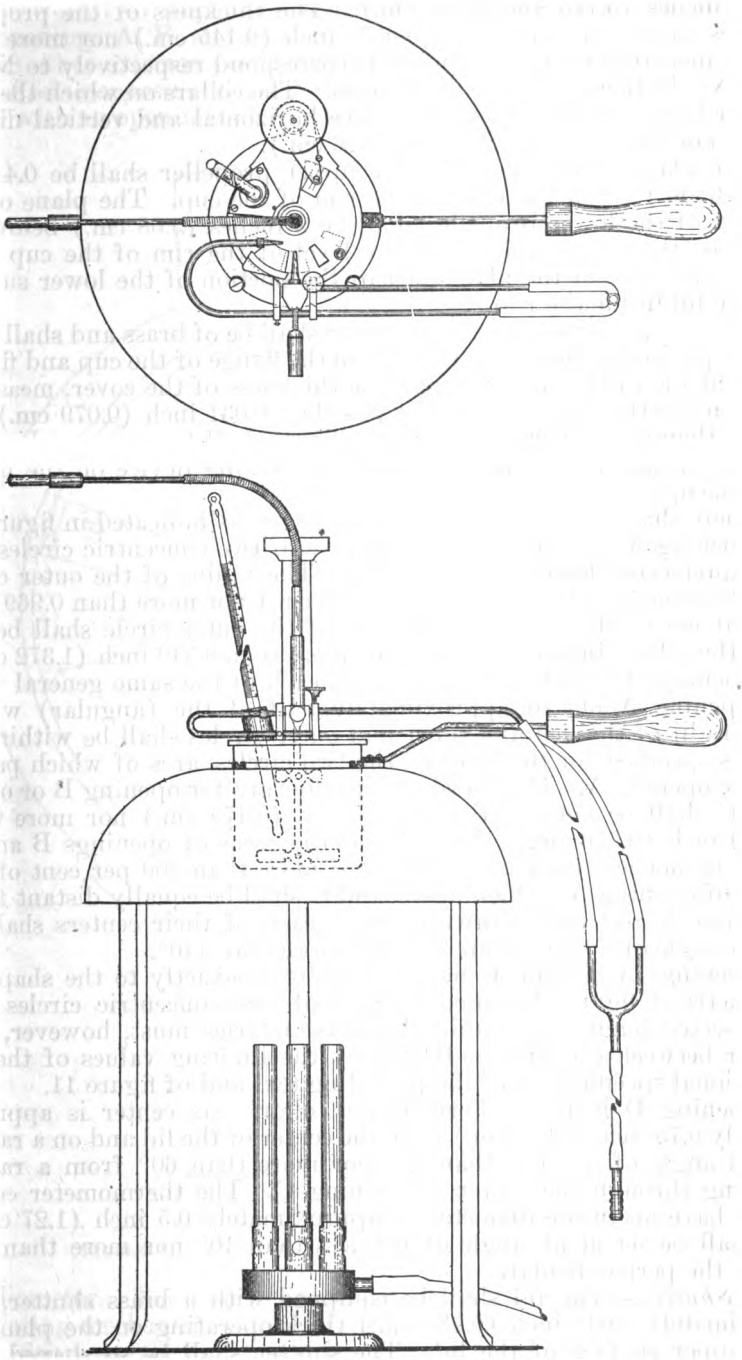

FIGURE 10.—Pensky-Martens closed tester (A. S. T. M.).

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(lower) propeller shall have an over-all diameter between 1.25 and 1.75 inches (3.175 and 4.445 cm.). The thickness of the propeller blades shall be not less than 0.057 inch (0.145 cm.) nor more than 0.081 inch (0.206 cm.) which limits correspond respectively to No. 15 and No. 12 B. and S. gage sheet brass. The collars on which the propeller blades are mounted shall have horizontal and vertical dimensions not greater than 0.4 inch (1.016 cm.).

The plane of the center of the upper propeller shall be 0.4 inch (1.016 cm.) below the level of the rim of the cup. The plane of the center of the lower propeller shall be 2 inches (5.08 cm.) below the level of the rim of the cup. The level of the rim of the cup is in effect the level of the plane part of the portion of the lower surface of the lid inside the rim.

2. *Cover proper.*—The cover proper shall be of brass and shall have a rim projecting downward almost to the flange of the cup and fitting the outside of the cup closely. The thickness of the cover, measured just inside the rim, shall not be less than 0.031 inch (0.079 cm.) nor more than 0.078 inch (0.198 cm.). There shall be a proper locating device engaging with a corresponding locating device on the flange of the cup.

There shall be four openings in the cover, as indicated in figure 11.

Opening A is an area defined by arcs of two concentric circles and the intersected lengths of two radii. The radius of the outer circle shall be not less than 0.938 inch (2.383 cm.) nor more than 0.969 inch (2.461 cm.). The chord of the arc of the outer circle shall be not less than 0.500 inch (1.270 cm.) nor more than 0.540 inch (1.372 cm.).

Openings B and C are equal areas, each of the same general form as opening A but of approximately half of the (angular) width. The radii of the defining inner and outer circles shall be within the limits specified for the radii of the two circles, arcs of which partly define opening A. The chord of the outer arc for opening B or opening C shall be not less than 0.187 inch (0.475 cm.) nor more than 0.219 inch (0.556 cm.). The sum of the areas of openings B and C shall be not less than 75 per cent nor more than 100 per cent of the area of opening A. Openings B and C shall be equally distant from opening A and radii drawn through each of their centers shall be at an angle of not less than 135° nor more than 140°.

Openings A, B, and C need not conform exactly to the shape of geometrical figures bounded by arcs of two concentric circles and intersected lengths of radii. Their boundaries must, however, fall on or between the lines indicated by the limiting values of the dimensional specification of the preceding text and of figure 11.

Opening D is for a thermometer collar. Its center is approximately 0.75 inch (1.905 cm.) from the center of the lid and on a radius at an angle of not less than 50° nor more than 60° from a radius passing through the center of opening C. The thermometer collar shall have an inside diameter of approximately 0.5 inch (1.27 cm.). It shall be set at an angle of not less than 10° nor more than 15° from the perpendicular.

3. *Shutter.*—The lid shall be equipped with a brass shutter, approximately 0.094 inch (0.239 cm.) thick operating on the plane of the upper surface of the lid. The shutter shall be so shaped and

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mounted that it rotates on the axis of the horizontal center of the lid between two stops so placed that when in one extreme position the openings A, B, and C of the lid are completely closed and when in the other extreme position these orifices are completely opened.

4. *Flame-exposure device.*—The flame-exposure device shall have a tip with an opening 0.027 inch (0.069 cm.) to 0.031 inch (0.079 cm.)

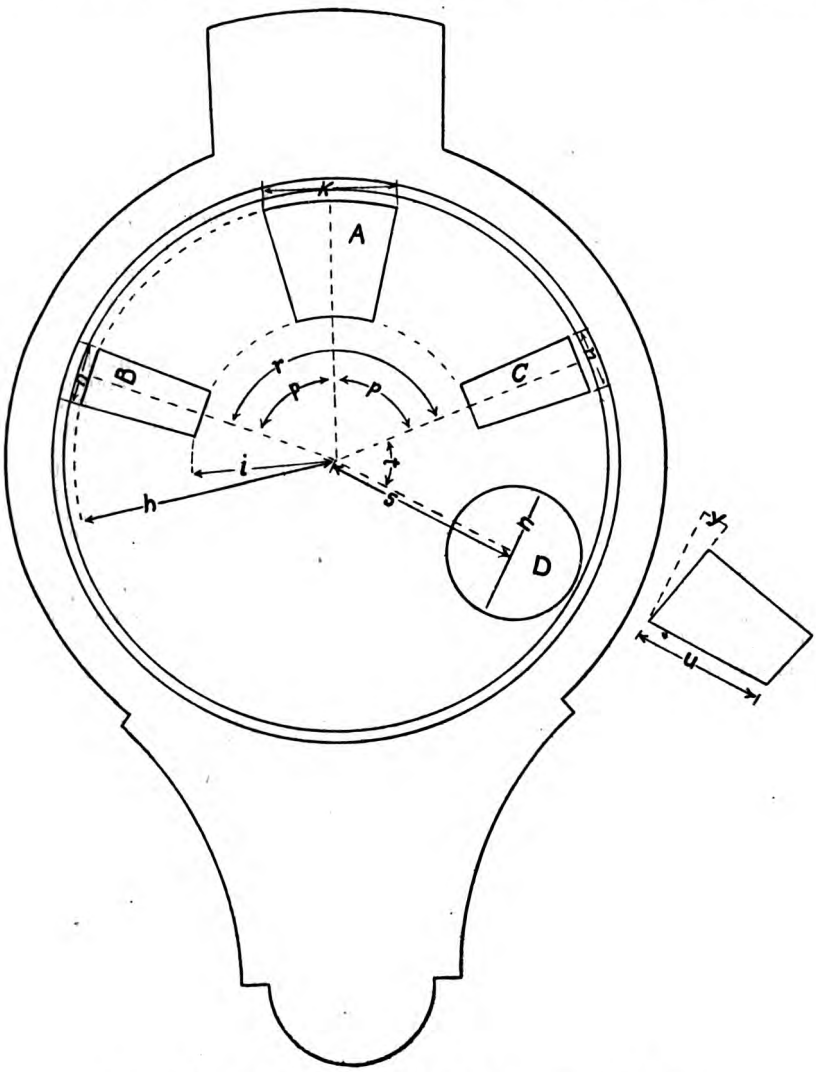

FIGURE 11.—Cover for Pensky-Martens tester (A. S. T. M.).

in diameter. The flame-exposure device shall be equipped with an operating mechanism which, when the shutter is in the "open" position, depresses the tip so that the center of the orifice is between the planes of the under and upper surfaces of the lid proper at a point on a radius passing through the center of the larger opening A and

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approximately 0.1 inch (0.254 cm.) from the outer edge of the opening.

NOTE.—A pilot flame for automatic relighting of the exposure flame should be provided. A bead $\frac{5}{32}$ inch in diameter, of some suitable material, may be mounted on the lid so that the size of the test flame can be regulated by comparison.

The mechanism operating the shutter should be of the spring type and so constructed that when at rest the shutter shall exactly close the three openings. When operated to the other extreme the three openings in the lid shall be exactly open and the tip of the exposure tube shall be fully depressed.

(*c*) *Stove.*—Heat shall be supplied to the cup by means of a properly designed stove which is equivalent to an air bath. This stove shall consist of (1) an air bath, and (2) a top plate on which the flange of the cup rests.

1. *Air bath.*—The air bath shall have a cylindrical interior 1.625 inches (4.128 cm.) to 1.656 inches (4.206 cm.) deep and a diameter not less than 0.125 inch (0.317 cm.) nor more than 0.156 inch (0.396 cm.) greater than the outside diameter of the cup. The air bath may be either a flame-heated metal casting or an electric resistance element.

NOTE.—If the heating element is a flame-heated metal casting it shall be so designed and used that the temperature of bottom and walls is approximately the same. On this account it should be not less than 0.25 inch (0.635 cm.) thick. The casting shall be so designed that products of combustion of the flame can not pass up and in contact with the cup.

If the air bath is of the electric-resistance type it shall be so constructed that all parts of the interior surface are heated equally. This necessitates an even distribution of resistance wire over bottom and walls and a method of construction such that heat is given out from the whole core of the resistance element rather than directly from the wire.

2. *Top plate.*—The top plate shall be of metal. The total distance from the upper surface of the plate to the bottom of the air bath shall exceed the distance from the under surface of the flange to the bottom of the cup by not less than 0.063 inch (0.160 cm.) nor more than 0.125 inch (0.317 cm.).

The top plate shall be mounted with an air gap between it and the air bath. The top plate may be attached to the air bath by means of three screws and spacing bushings. The spacing bushings should be of proper thickness to define the air gap, which shall be not less than 0.125 inch (0.317 cm.) nor more than 0.187 inch (0.475 cm.). The spacing bushings shall be not more than 0.375 inch (0.952 cm.) in diameter.

(*d*) *Thermometers.*—Two standard thermometers shall be used with the A. S. T. M. Pensky-Martens tester. The low range, "P. M. and Tag" thermometers shall be used for tests when the indicated reading falls within the limits 20° to 200° F. The "P. M. high" thermometer shall be used for tests when the indicated reading falls within the limits 230° to 700° F. For the range 200° to 230° F. either thermometer may be employed, depending on the convenience of the operator. The thermometers shall comply with the specifications given in Table 1.

Thermometers shall be so mounted that the bottom of the bulb is 1.75 inches (4.445 cm.) below the level of the rim of the cup (which corresponds to the level of the lower surface of the portion of the lid inside the rim.)

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TABLE 1.—*Specifications for thermometers for Pensky-Martens tester.*

	Low range "P. M. and Tag."[a]	High range "P. M. high."
Type	Etched stem glass	Etched stem glass.
Total length	275 mm	275 mm.
Stem	Plain front, enamel back, suitable thermometer tubing; diameter, 6 to 7 mm.	Plain front, enamel back, suitable thermometer tubing; diameter, 6 to 7 mm.
Bulb	Corning normal, Jena 16 III, or equally suitable thermometric glass; diameter, less than stem; length, 9 to 13 mm.	Corning normal, Jena 16 III, or equally suitable thermometric glass; diameter, less than stem; length, maximum, 10 mm.
Actuating liquid	Mercury	Mercury.
Range	20° F. to 230° F	200° F. to 700° F.
Immersion	2¼ inches (57 mm.) from end of bulb. Words "2¼ in. Immersion" etched on the stem, also a line around the stem to indicate depth of immersion.	2¼ inches (57 mm.) from end of bulb. Words "2¼ in. Immersion" etched on the stem, also a line around the stem to indicate depth of immersion.
Distance to 20° line from bottom of bulb.	75 to 90 mm	
Distance to 200° line from bottom of bulb.		75 to 90 mm.
Distance to 230° line from top to stem.	25 to 40 mm	
Distance to 700° line from top of stem.		25 to 40 mm.
Expansion chamber	Required	None.
Filled	Nitrogen gas	Nitrogen gas.
Top finish	Glass ring	Glass ring.
Graduating	All lines, figures, and letters clear cut and distinct; scale graduated in 1° divisions; scale numbered every 10°, the first and each succeeding 5° line to be longer than the others.	All lines, figures, and letters clear cut and distinct; scale graduated in 5° divisions; scale numbered every 50°, the first and each succeeding 25° line to be longer than the others.
Special marking	"A. S. T. M., P. M., and Tag," serial number, manufacturer's name or trade-mark etched on the stem.	"A. S. T. M., P. M., high," serial number, manufacturer's name or trade-mark etched on the stem.
Accuracy	Error at any point in scale shall not exceed 1°.	Error at any point in scale shall not exceed one-half smallest scale division.
Test for permanency		After being subjected to a temperature of 680° F. for 24 hours the accuracy shall be within the limit specified.
Points to be tested	32°, 100°, 150°, 212° F	212°, 450°, 700°F.

[a] The low range "P. M. or Tag" thermometer is the instrument specified for use with the Tag closed tester.

PROCEDURE.

3. (*a*) All parts of the cup and its accessories shall be thoroughly clean and dry before the test is started. Particular care shall be taken to avoid the presence of gasoline or naphtha used to clean the apparatus after a previous test.

(*b*) The cup shall be filled with the oil to be tested up to the level indicated by the filling mark.

(*c*) The lid shall be placed on the cup and the latter set in the stove. Care shall be taken to have the locating devices properly

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engaged. The thermometer shall be inserted. If it is known that the oil will flash above 220° F. the "P. M. High" thermometer may be selected; otherwise, it is preferable to start with the "P. M. and Tag" thermometer and change in case a temperature of 220° to 230° F. is reached.

(*d*) The test flame shall be lighted and so adjusted that it is of the size of a bead $\frac{5}{32}$ of an inch in diameter.

(*e*) Heat shall be supplied at such a rate that the temperature read on the thermometer increases not less than 9° nor more than 11° F. per minute. The stirrer shall be turned at a rate of from one to two revolutions per second.

(*f*) Application of the test flame shall be made at each temperature reading which is a multiple of 2° F. up to 220° F. For the temperature range above 220° F., application shall be made at each temperature reading which is a multiple of 5° F. Application of the test flame shall be made by operating the device controlling the shutter and test flame burner so that the flame is lowered in one-half second, left in its lowered position for one second, and quickly raised to its high position. Stirring shall be discontinued during the application of the test flame.

4. The flash point is taken as the temperature read on the thermometer at the time of the flame application that causes a distinct flash in the interior of the cup. The true flash must not be confused with the bluish halo that sometimes surrounds the test flame for the applications preceding the one that causes the actual flash.

5. The barometric pressure shall be observed and recorded. No corrections shall be made except in case of dispute, when the flash-point figure shall be corrected according to the following rule:

For each inch (25 mm.) below 29.92 inches (760 mm.) barometric reading add 1.6° F. to the flash point.

For each inch (25 mm.) above 29.92 inches (760 mm.) barometric reading subtract 1.6° F. from the flash point.

METHOD 110.3—OPEN CUP FLASH AND FIRE TEST.

[A. S. T. M. Method D92–21T.]

1. The open cup flash and fire test on all petroleum products except fuel oils and those having an open cup flash below 175° F. shall be determined in the Cleveland open cup.

APPARATUS.

2. The flash cup proper shall be made of brass and shall have the following dimensions:

Specifications of flash cup.

	Dimensions.	Tolerances.	Dimensions.	Tolerances.
	Inches.	*Inches.*	*Cm.*	*Cm.*
Inside diameter	$2\frac{1}{2}$	$\pm\frac{1}{32}$	6.350	±0.079
Outside diameter	$2\frac{1}{16}$	$\pm\frac{1}{32}$	6.826	± .079
Inside height	$1\frac{5}{16}$	$\pm\frac{1}{32}$	3.334	± .079
Thickness of bottom	$\frac{1}{8}$	$\pm\frac{1}{64}$	.318	± .040
Depth of filling mark below top of cup	$\frac{3}{8}$	$\pm\frac{1}{64}$	.953	± .040

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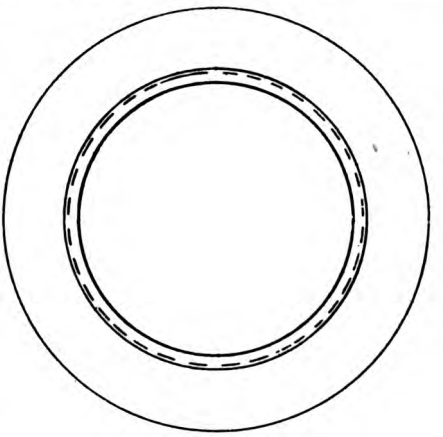
The cup shall be heated by contact with a metal plate $\frac{1}{4}$ inch (0.635 cm.) thick and 6 inches (15.24 cm.) wide. (The plate may be of any suitable metal and may be either circular or square.) In the center of the plate there shall be a plane depression $\frac{1}{32}$ inch (0.079 cm.) deep and of just sufficient diameter to fit the cup. The plate shall be covered with a sheet of hard asbestos board $\frac{1}{4}$ inch thick and of the same shape as the metal plate. There shall be cut in the center of the asbestos board a circular hole just fitting the cup. The metal plate may be heated in any convenient manner. The use of a gas burner, electric heater, or alcohol lamp is permitted. If a flame heater is used it may be protected from drafts or excessive radiation by any suitable type of shield that does not project above the level of the upper surface of the asbestos board.



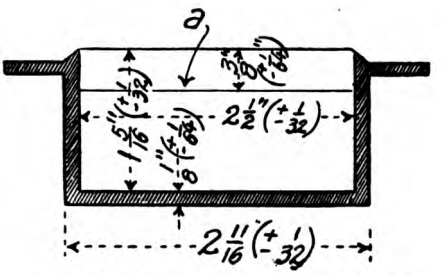


FIGURE 12.—Cleveland open cup (A. S. T. M.): *a*, Filling mark.

3. The thermometer shall conform to the following specifications:

Type: Etched stem glass.

Total length: 305 mm.

Stem: Plain front, enamel back, suitable thermometer tubing; diameter, 6 to 7 mm.

Bulb: Corning normal, Jena 16 III, or equally suitable thermometric glass; length, 13 mm., maximum; diameter, not greater than stem.

Actuating liquid: Mercury.

Range: +20° to +760° F.

Immersion: 1 inch. The words "1-inch Immersion" shall be etched on the stem and also line around the stem to indicate the depth of immersion.

Distance to 20° line from bottom of bulb: 40 to 50 mm.

Distance to 760° line from top of stem: 30 to 45 mm.

Filled: Nitrogen gas.

Top finished: Red glass ring.

Graduation: All lines, figures, and letters clear cut and distinct; scale graduated in 5° divisions; scale numbered every 20°, the first and each succeeding 10° F. line to be longer than the others.

Special marking: "A. S. T. M. Open Flash," serial number, and manufacturer's name or trade-mark etched on stem.

Accuracy: Error at any point on scale shall not exceed one-half smallest scale division up to 700° F.

Test for permanency of range: After being subjected to a temperature of 700° F. for 24 hours, the accuracy shall be within the limit specified.

Points to be tested for certification: 32°, 212°, 400°, 700° F.

PROCEDURE.

4. (*a*) The thermometer shall be suspended or held in a vertical position by any suitable device. The bottom of the bulb shall be

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$\frac{1}{4}$ inch [6] from the bottom of the cup and above a point half way between the center and back of the cup.

(*b*) The cup shall be filled with the oil to be tested in such a manner that the top of the meniscus is exactly at the filling line at room temperature. The surface of the oil shall be free from bubbles. There shall be no oil above the filling line or on the outside of the apparatus.

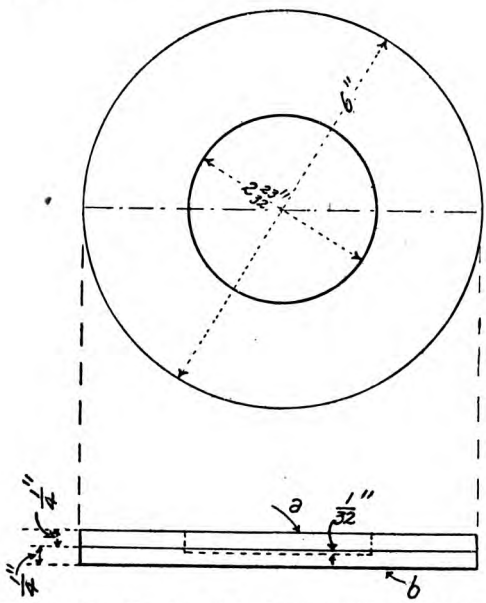

FIGURE 13.—Heating plate for use with Cleveland open cup (A. S. T. M.): *a*, Asbestos disk; *b*, plate.

(*c*) The test flame shall be approximately $\frac{5}{32}$ inch in diameter.

NOTE.—For purposes of comparison it is recommended that a bead of suitable light-colored material be mounted in a convenient position so that the size of the test flame can be determined. The device for applying the flame may be of any suitable type, but it is suggested that the tip be approximately $\frac{1}{16}$ inch in diameter at the end and that the orifice be $\frac{1}{32}$ inch in diameter. If the device for operating the test flame be mounted in such a manner as to permit automatic duplication of the sweep of the test flame the radius of swing shall be not less than 6 inches.

(*d*) The test flame shall be applied as the temperature read on the thermometer reaches each successive 5° F. mark. The flame shall pass in a straight line (or on the circumference of a circle having a radius of at least 6 inches) across the center of the cup and at right angles to the diameter passing through the thermometer. The test flame shall, while passing across the surface of the oil, be in the plane of the upper edge of the cup. The time for the passage of the test flame across the cup shall be approximately one second.

(*e*) The rate of heating of the oil shall be such that the temperature read on the thermometer increases not less than 9° nor more than 11° F. per minute.

5. The flash point shall be taken as the temperature read on the thermometer when a flash appears at any point on the surface of the oil. The true flash must not be confused with a bluish halo that sometimes surrounds the test flame.

6. After determining the flash point the heating shall be continued at the specified rate, and application of the test flame shall be made at the specified intervals until the oil ignites and continues to burn

[6] The immersion line engraved on the thermometer stem will be $\frac{1}{16}$ inch below the level of the rim of the cup.

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for a period of at least five seconds. The temperature read when this occurs shall be taken as the fire point.

7. The flash point and fire point tests shall be made in a room or compartment free from air drafts. The operator shall avoid breathing over the surface of the oil. It is desirable that the room or compartment be darkened sufficiently to make the flash readily discernible.

METHOD 120.1.—SPOT TEST.

[From Bureau of Standards Circular 98.]

Place 5 drops of the oil on clean white filter paper and allow the liquid to evaporate at room temperature, away from direct sunlight. There should be no oily spot left after 30 minutes.

METHOD 130.1.—FLOCK TEST FOR KEROSENE.

APPARATUS AND PROCEDURE.

Place 300 c. c. of the sample in a 500 c. c. Florence or Erlenmeyer flask (after filtering if it contains suspended matter). Suspend a thermometer in the oil by means of a cork slotted on the side, and place the flask containing the oil in a sand bath, or on an electric hot plate. Heat at such a rate that the oil will reach a temperature of 240° F. at the end of one hour. Hold oil at a temperature of not less than 240° F. nor more than 250° F. for six hours. Give the flask a slight rotary motion, and if any flock is seen rising from the bottom the test shall be reported as positive. Discoloration of the oil shall be disregarded.

METHOD 130.2.—FLOCK TEST FOR MINERAL SEAL OIL.

APPARATUS AND PROCEDURE.

Place 300 c. c. of the sample in a 500 c. c. Florence or Erlenmeyer flask (after filtering if it contains suspended matter). Suspend a thermometer in the oil by means of a cork slotted on the side, and place the flask containing the oil in a sand bath, or on an electric hot plate. Heat the oil at the rate of 10° F. per minute to a temperature of 450° F. and hold at that temperature for 15 minutes. Give the flask a slight rotary motion, and if any flock is seen rising from the bottom the test shall be reported as positive. Allow the flask to cool for one hour and again examine for flock. Discoloration of the oil shall be disregarded.

METHOD 200.1.—WICK-FEED TEST.

APPARATUS.

1. An oil container, made of brass, capacity about 1 quart, fitted in the center with a brass tube of $\frac{1}{2}$ inch internal diameter, which serves as an oilway and which feeds into a graduated glass cylinder.

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2. A wick composed of eight strands of worsted zephyr of the best quality, pure, long fiber, cream white fine wool, thoroughly washed, scoured, and carded; not dyed nor subjected to any chemical process.

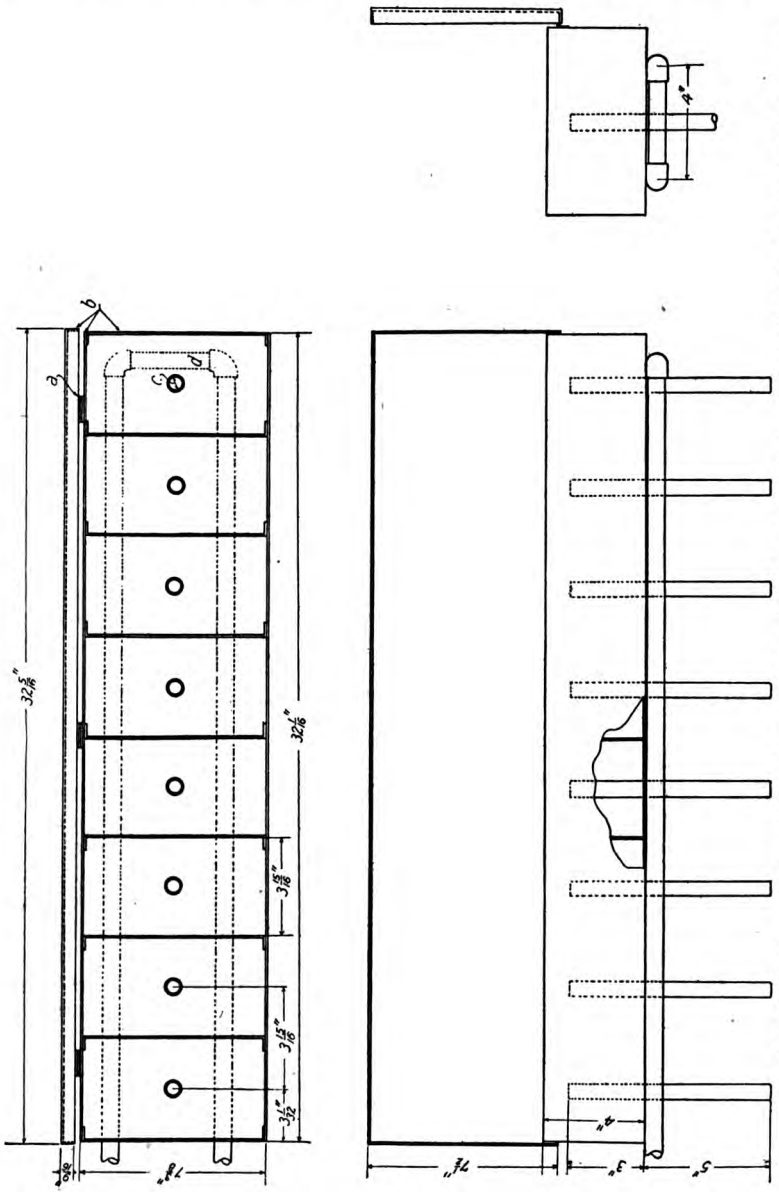

FIGURE 14.—Wick-feed test apparatus, as used by the U. S. Naval Engineering Experiment Station, Annapolis, Md.: *a*, Queen inch brass hinge; *b*, 1/16-inch sheet brass; *c*, 1/4-inch (inside diameter) brass tube; *d*, 1/8-inch steam pipe.

Strands shall be four-ply soft spun and twisted. The separate plies shall be of uniform thickness throughout their entire length.

3. A wick support in the form of a hook of copper wire which grips the outlet end of the wick below the level of the oil, as is the usual manner in a wick feed.

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PROCEDURE.

4. Dip the wick in the oil and place it in position and fill the oil container so that the lift of the wick is $\frac{1}{4}$ inch. Maintain the lift at from $\frac{1}{2}$ to $\frac{1}{4}$ inch during the test. Measure the flow of oil at the end of 24 hours and again at the end of 14 days.

METHOD 210.1.—BURNING TEST FOR KEROSENE (18 HOURS).

APPARATUS.

A lamp fitted with a No. 1 sun hinge burner.

PROCEDURE.

Place a clean, dry No. 1 wick in the burner and fill the lamp with oil to be tested, adjust the chimney, light the lamp, and adjust the wick so that the lamp burns with a flame not over 1 inch high. At the end of one-half hour again adjust the wick so that the largest possible flame is obtained without smoking.

Allow the lamp to burn continuously for 18 hours.

At the end of this time the flame must be clear and practically as large as when the final adjustment of the wick was made. No accumulations of soot or other material shall be formed on the wick during the test and the chimney must be clear or only slightly clouded.

METHOD 210.2.—BURNING TEST FOR KEROSENE (8 HOURS).

APPARATUS.

A lamp fitted with a No. 1 sun hinge burner.

PROCEDURE.

Place a clean, dry No. 1 wick in the burner and fill the lamp with oil to be tested, adjust the chimney, light the lamp, and adjust the wick so that the lamp burns with a flame not over 1 inch high. At the end of one-half hour again adjust the wick so that the largest possible flame is obtained without smoking.

Allow the lamp to burn continuously for eight hours.

At the end of this time the flame must be clear and practically as large as when the final adjustment of the wick was made. No accumulations of soot or other material shall be formed on the wick during the test and the chimney must be clear or only slightly clouded.

METHOD 210.3—BURNING TEST FOR LONG-TIME BURNING OIL.

APPARATUS.

A standard Railway Signal Association semaphore lamp, fitted with the puchaser's standard burner, chimney, and wick.

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PROCEDURE.

Place a new wick, which has been washed with redistilled ether and dried at room temperature, in the burner. Place the lamp in a position protected from the direct rays of the sun, either out of doors or in a well-ventilated room, fill it with the oil to be tested, light it, and adjust the wick so as to produce a flame $\frac{3}{4}$ inch high, measured from the top of the wick; readjust if necessary during the first four hours. Allow the lamp to burn without readjusting the wick for 120 hours. The flame shall remain symmetrical and free from smoke throughout the test period, the height of flame at no time during the test shall be less than $\frac{5}{8}$ inch and no appreciable hard incrustation shall be formed on the wick.

METHOD 210.4.—BURNING TEST FOR MINERAL SEAL OIL.

APPARATUS.

A lamp fitted with a dual burner No. 3, dual chimney, and duplex wicks. The distance from the top of the wicks to the bottom of the inside of the font shall be not less than $6\frac{1}{2}$ inches nor more than 7 inches.

PROCEDURE.

Place 20 ounces of the oil to be tested in the lamp, light it, and adjust the wicks so as to produce a symmetrical flame approximately 1 inch high, measured from the top of the wicks; readjust if necessary during the first hour. The lamp shall then be burned continuously without readjusting the wicks until all the oil is consumed. The flame shall remain symmetrical and free from smoke throughout the test period. No appreciable hard incrustation shall be formed on the wick.

METHOD 210.5.—BURNING TEST FOR SIGNAL OIL.

APPARATUS.

A standard railway signal hand lantern, with an 8-ounce font and a burner fitted with a 1-inch wick.

PROCEDURE.

Place the oil to be tested in the lantern and adjust the wick to give a satisfactory flame. The test shall be continued for 24 hours without trimming or adjusting the wick, refilling the lantern if necessary. A satisfactory flame must be maintained throughout the test period; no appreciable amount of hard incrustation shall be formed on the wick. *The flame must stand all forms of railroad signaling in any kind of weather without being extinguished or smoking the globe.*

METHOD 300.1.—WATER.

[A. S. T. M. Method D95–21T.]

1. This method shall be used for crude petroleum, fuel oil, and road oil, and may be used for any petroleum product.

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SAMPLE.

2. The sample shall be thoroughly representative of the material to be tested, and the portion of the sample used for the test shall be thoroughly representative of the sample itself. Deviation from this rule shall not be permitted. The difficulties in obtaining proper representative samples for this determination are unusually great, so that the importance of sampling can not be too strongly emphasized.

APPARATUS.

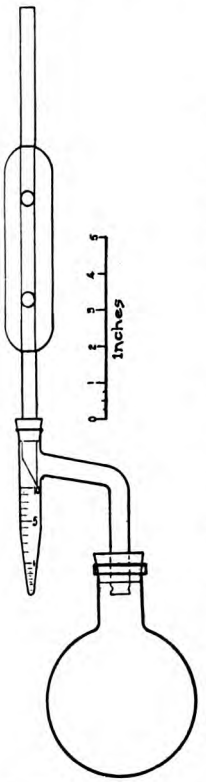

FIGURE 15.—Apparatus for determination of water in petroleum products (A. S. T. M.).

3. The preferred form shall be that of Dean and Stark, as shown in figure 15, and shall include the following:

(*a*) Distillation flask, 500-c. c. capacity, of copper or well-annealed glass, round bottom.

(*b*) Reflux condenser, water-cooled glass-tube type.

(*c*) Special graduated distilling-tube receiver of well-annealed glass, graduated upward from 0 to 10 c. c. in 0.1 c. c. divisions, accurate to 0.05 c. c.

(*d*) Gas burner or suitable electric heater.

4. An optional form may be used and shall include the following:

(*a*) Distillation flask, 500-c. c. capacity.

(*b*) Condenser, water-cooled glass-tube, free from traps retarding complete draining.

(*c*) Receiver, of glass, capacity about 125 c. c., A. S. T. M. water and sediment tube or equivalent. The A. S. T. M. water and sediment tube is shown in figure 17, and is graduated upward from 0 to 100 c. c. as follows:

Graduation of A. S. T. M. water and sediment tube.

Range.	Scale divisions.	Limit of error.	Numbered.
C. c.	*C. c.*	*C. c.*	*C. c.*
0– 3	0. 1	0. 05	1, 2, 3.
3– 5	0. 5	. 2	4, 5.
5– 10	1. 0	. 5	6, 8, 10.
10– 25	5. 0	1. 0	15, 20, 25.
50–100	50. 0	1. 0	50, 100.

(*d*) Gas burner or suitable electric heater.

(*e*) Thermometer, engraved-stem type, to indicate 400° F.

5. The diluent used in this method shall be gasoline, free from water. When subjected to distillation (A. S. T. M. method D86–21T) it shall show 5 per cent at a temperature not above 212° F. (100° C.) nor below 194° F. (90° C.) It shall show 90 per cent at a temperature not above 410° F. (205° C.).

PROCEDURE.

6. Exactly 100 c. c. of the oil to be tested shall be measured in an accurate 100 c. c. graduated cylinder at room temperature and poured

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into the distillation flask. The oil adhering to the walls of the 100 c. c. graduated cylinder shall be transferred to the distillation flask by rinsing with two successive 50-c.c. portions of gasoline, the cylinder being allowed to drain each time. The oil and gasoline in the distillation flask shall be thoroughly mixed by swirling the flask with proper care to avoid any loss of material. A boiling stone, such as a piece of unglazed porcelain, may be introduced for the purpose of preventing bumping during the subsequent distillation.

7. The apparatus shall be assembled in the following manner:

(*a*) If the preferred form of apparatus is used, the distillation flask shall be connected with the special graduated distilling tube receiver and this receiver with the reflux condenser by means of tight-fitting corks, as shown in figure 15.

(*b*) If the optional form of apparatus is used, the vapor outlet tube of the distillation flask shall be inserted into the condenser, a tight connection being made by means of a cork. The receiver shall be supported in a suitable position at the other end of the condenser without the use of a cork or other connection. Proper precautions shall be taken to prevent the introduction into the receiver of water from any source other than the subsequent distillation process.

The mouth of the distillation flask shall be closed by a tight-fitting cork supporting the thermometer in such a position that the top of the thermometer bulb is level with the bottom of the vapor outlet tube, where the latter is joined to the neck of the flask.

8. Either form of apparatus being assembled as prescribed, heat shall be applied to the distillation flask and shall be so regulated that the condensed distillate falls from the end of the condenser at the rate of 2 to 4 drops per second.

9. (*a*) With the preferred form of apparatus, distillation shall be continued at the specified rate until no water is visible on any part of the apparatus except at the bottom of the distilling tube receiver. This operation usually requires less than one hour. A persistent ring of condensed water in the condenser tube shall be removed by increasing the rate of distillation for a few minutes.

(*b*) With the optional form of apparatus, distillation shall be continued at the specified rate until the thermometer indicates a vapor temperature of 400° F. and until all condensed water has disappeared from the walls of the distillation flask and of the condenser.

10. With either form of apparatus, the volume of condensed water, measured in the graduated receiver at room temperature, shall be recorded as "— per cent water, A. S. T. M. method."

ACCURACY.

11. With proper care and attention to details, duplicate determinations of water by this method shall not differ from each other by more than 0.2 c. c. of water, provided the graduated receiver is accurate and readable to this degree.

NOTE.—When the sample to be tested contains more than 10 per cent of water, the volume of material used shall be decreased to that which will yield somewhat less than 10 c. c. of water. Otherwise the procedure shall be conducted as prescribed.

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METHOD 300.2.—SEDIMENT.

APPARATUS.

Alundum (porous grade) thimbles,[7] 1 inch in diameter by $2\frac{3}{4}$ inches high, weighing not less than 15 nor more than 17 grams.

Extraction apparatus (see fig. 16) of such construction that the thimble is completely surrounded by the vapor of the boiling solvent. *Syphon extractors must not be used.*

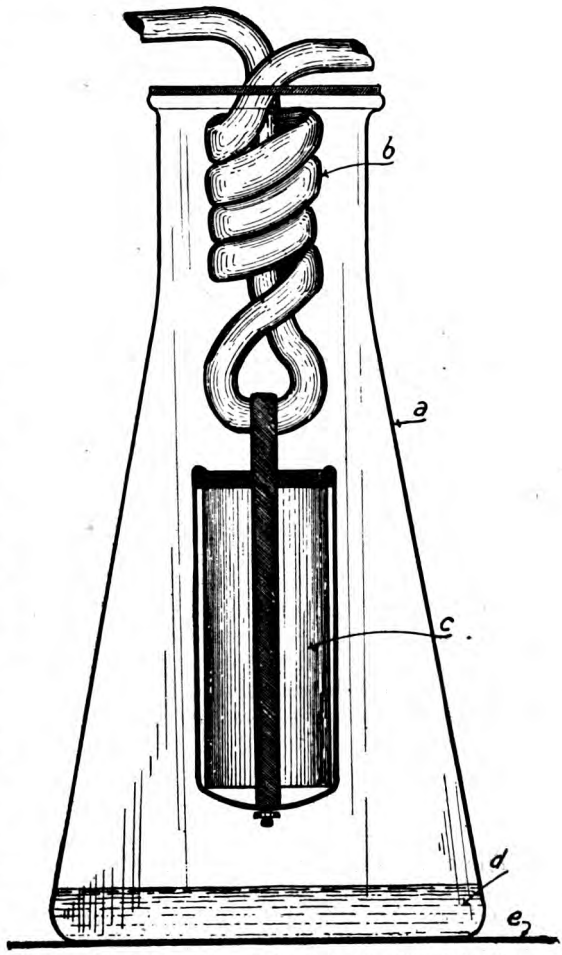

FIGURE 16.—Extraction apparatus for determination of sediment: *a*, Extraction flask; *b*, condenser; *c*, extraction thimble; *d*, solvent; *e*, top of hot plate.

PROCEDURE.

Place approximately 10 grams of the sample in the previously extracted and dried, accurately weighed thimble and weigh to ±0.01 gram, place in the extraction apparatus, and extract with 90 per cent benzol until the solvent dropping from the thimble is colorless. Dry the thimble for one hour at 105° C. and weigh to ±0.0001 gram. Repeat the extraction until the weight of the dried thimble and sediment is constant.

NOTE.—The rate of extraction shall be such that the mixture of oil and benzol in the thimble does not rise to within one-quarter inch of the top.

METHOD 300.3.—WATER AND SEDIMENT BY CENTRIFUGE.

[A. S. T. M. Method D96–21T.]

1. This method may be used for crude mineral oils and fuel oils. A centrifuge method for "Water and sediment" is not entirely satisfactory because the amount of water obtained is nearly always lower

[7] Alundum thimbles listed by the Norton Co., Worcester, Mass., as 5163 RA 98, have proven satisfactory.

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than the actual water content. Nevertheless, on account of the wide use of the centrifuge for this purpose, it is desirable that the method of making the determination be standardized as far as possible. It must be clearly understood that the reading of the centrifuge tube includes both the sediment and the precipitated water. Accurate determination of water content, if desired, should be made in accordance with the "Tentative Method of Test for Water in Petroleum Products (D95–21T)" of the American Society for Testing Materials (Method 300.1 above).

2. The sample shall be thoroughly representative of the material in question and the portion used for the test shall be thoroughly representative of the sample itself. Deviation from this rule shall not be permitted. The difficulties in obtaining representative samples for this determination are unusually great; hence the importance of sampling can not be too strongly emphasized.

APPARATUS.

3. The centrifuge shall be capable of whirling at the required speed at least two 100-c. c. centrifuge tubes filled with water. It shall be of sound design and rugged construction so that it may be operated without danger. The tube carriers shall be so designed that the glass centrifuge tubes may be cushioned with water, rubber, or other suitable material. The tube holders shall be surrounded during the operation by a suitable metal shield or case, strong enough to eliminate danger if any breakage occurs.

The preferred form of centrifuge shall have a diameter of swing (tip to tip of whirling tubes) of 15 to 17 inches and a speed of at least 1,500 r. p. m. or the equivalent. If the available centrifuge has a diameter of swing varying from these limits, it shall be run at the proper speed to give the same centrifugal force at the tips of the tubes as that obtained with the preferred form of centrifuge. The proper speed shall be calculated from the following formula in which d represents diameter of swing (tip to tip of whirling tubes) of the centrifuge used:

$$r.\ p.\ m. = 1500\sqrt{\frac{16}{d}}.$$

4. The centrifuge tubes, A. S. T. M. type, shall be made of suitable glass and thoroughly annealed. The total capacity shall be about 125 c. c. and the mouth shall be suitably constricted for closing with a cork. The graduations shall be clear and distinct, reading upward from the bottom of the tube as follows:

Graduations of centrifuge tubes.

Range.	Scale of divisions.	Limit of error.	Numbered.
C.c.	*C. c.*	*C. c.*	*C. c.*
0– 3	0.1	0.05	1, 2, 3.
3– 5	.5	.2	4, 5.
5– 10	1.0	.5	6, 8, 10.
10– 25	5.0	1.0	15, 20, 25.
50–100	50.0	1.0	50, 100.

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The shape is optional, provided it does not conflict with the other requirements. Satisfactory types are shown in figure 17.

5. The water or oil bath shall be of sufficient depth for immersing the centrifuge tubes in a vertical position to the 100-c. c. mark. Means shall be provided for heating this bath to 100° F.

PROCEDURE.

6. (*a*) Exactly 50 c. c. of 90 per cent benzol shall be measured into each of two centrifuge tubes and exactly 50 c. c. of the oil to be tested shall then be added to each. The centrifuge tubes shall be tightly stoppered and shall be shaken vigorously until the contents are thoroughly mixed. The temperature of the bath shall be maintained at 100° F., and the centrifuge tubes shall be immersed therein to the 100-c. c. mark for 10 minutes.

(*b*) The two centrifuge tubes shall then be placed in the centrifuge on opposite sides and shall be whirled at a rate of 1,400 to 1,500 r. p. m. or the equivalent for 10 minutes. The combined volume of water and sediment at the bottom of each tube shall be read and recorded, estimating to 0.1 c. c. if necessary. The centrifuge tubes shall then be replaced in the centrifuge, again whirled for 10 minutes, and removed for reading the volume of water and sediment as before. This operation shall be repeated until the combined volume of water and sediment in each tube remains constant for three consecutive readings. In general, not more than four whirlings will be required.

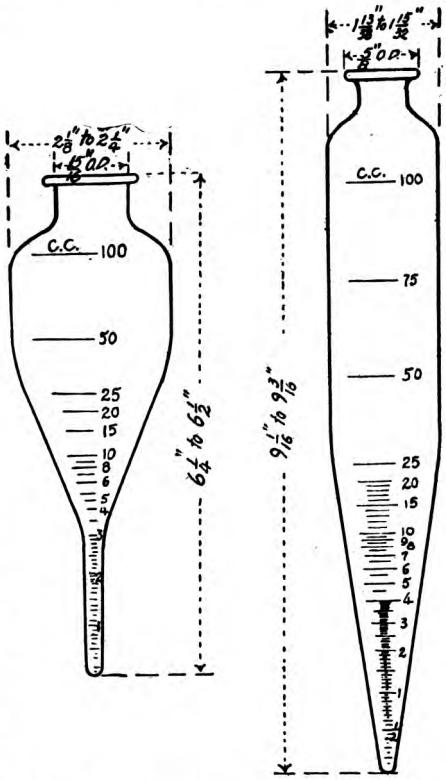

FIGURE 17.—Centrifuge tubes (A. S. T. M.)

7. The combined total volume of water and sediment shall be read on each tube, estimating to 0.1 c. c. if necessary. The sum of the two readings shall be recorded as percentage of water and sediment, centrifuge method.

ACCURACY.

8. With care and proper attention to details, duplicate determinations of water and sediment by this method should not differ by more than 0.2 c. c., provided the centrifuge tubes are accurate and readable to this degree.

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METHOD 300.4—WATER BY CENTRIFUGE

APPARATUS.

1. The apparatus shall be the same as in method 300.3.

2. Petroleum naphtha meeting the following requirements shall be used for the diluent:

Specific gravity at 60° F., 0.695 to 0.705.
Initial boiling point, A. S. T. M. 113 to 131° F. (45 to 55° C.)
End point, A. S. T. M., not higher than 248° F. (120° C.)

PROCEDURE.

3. Exactly 20 c. c. of the prescribed diluent shall be measured into each of two centrifuge tubes and exactly 20 c. c. of the oil to be tested shall then be added to each. The centrifuge tubes shall be tightly stoppered and shall be shaken vigorously until the contents are thoroughly mixed.

4. The two centrifuge tubes shall then be placed in the centrifuge on opposite sides, and shall be whirled at a rate of 1400 to 1500 r. p. m. or the equivalent for 10 minutes. The volume of water at the bottom of each tube shall be read and recorded, estimating to 0.1 c. c. if necessary. The centrifuge tubes shall then be replaced in the centrifuge, again whirled for 10 minutes, as before, and removed for reading the volume of water, as before. This operation shall be repeated until the volume of water in each tube remains constant for three consecutive readings.

5. The volume of water shall be read on each tube, estimating to 0.1 c. c. if necessary, and the average percentage of water in the sample calculated.

METHOD 310.1—PRECIPITATION NUMBER.

[A. S. T. M. Method D91–21T.]

1. This method is commonly used for steam cylinder stocks and black oils, and may be used for other lubricating oils.

DEFINITION.

2. The A. S. T. M. precipitation number is the number of cubic centimeters of precipitate formed when 10 c. c. of lubricating oil are mixed with 90 c. c. of petroleum naphtha of definite quality and centrifuged under definite prescribed conditions.

APPARATUS.

3. *Centrifuge.*—The centrifuge shall be capable of whirling at least two 100 c. c. centrifuge tubes filled with water at the required speed. It shall be of sound design and rugged construction, so that it may be operated without danger. The tube carriers shall be so designed that the glass centrifuge tubes may be cushioned with water, rubber, or other suitable material. The tube holders shall be sur-

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rounded during the operation by a suitable metal shield or case strong enough to eliminate danger if any breakage occurs.

Preferred forms of centrifuge shall have a diameter of swing (tip to tip of whirling tubes) of 15 to 17 inches and a speed of at least 1,500 r. p. m. or equivalent. If the available centrifuge has a diameter of swing varying from these limits, it shall be run at the proper speed to give the same centrifugal force at the tips of the tubes as that obtained with the preferred form of centrifuge. The proper speed shall be calculated from the following formula, in which d represents the diameter of swing (tip to tip of whirling tubes) of the centrifuge used:

$$r.\ p.\ m. = 1{,}500\sqrt{\frac{16}{d}}$$

4. *Centrifuge tubes, A. S. T. M. type.*—These tubes shall be made of suitable glass and thoroughly annealed. The total capacity shall be about 125 c. c., and the mouth shall be suitably constricted for closing with a cork. The graduations shall be clear and distinct, reading upward from the bottom of tube, as follows:

Graduations of centrifuge tubes, A. S. T. M. type.

Range.	Scale divisions.	Limit of error.	Numbered.
C. c.	*C. c.*	*C. c.*	
0– 3	0.1	0.05	1, 2, 3.
3– 5	.5	.2	4, 5.
5– 10	1.0	.5	6, 8, 10.
10– 25	5.0	1.0	15, 20, 25.
50–100	50.0	1.0	50, 100.

The shape is optional provided it does not conflict with the other requirements.

5. Petroleum naphtha meeting the following requirements shall be used for the diluent:

Specific gravity at 60° F., 0.695–0.705.
Initial boiling point, A. S. T. M., 113–131° F. (45–55° C.).
End point, A. S. T. M., not higher than 248° F. (120° C.).

PROCEDURE.

6. Exactly 10.0 c. c. of the oil to be tested shall be measured in each of two clean dry centrifuge tubes at room temperature. Each tube shall be filled to the 100-c. c. mark with the prescribed diluent and closed tightly with a softened cork (not a rubber stopper). Each tube shall then be inverted at least 20 times, allowing the liquid to drain thoroughly from the tapered tip of the tube each time. The tubes shall then be placed in a water bath at 90° to 95° F. for five minutes. The corks shall be momentarily removed to relieve any pressure and each tube shall again be inverted at least 20 times, exactly as before. The success of this method depends to a large degree upon having a thoroughly homogeneous mixture which will drain quickly and completely from the tapered tip when the tube is inverted.

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7. The two centrifuge tubes shall then be placed in the centrifuge on opposite sides and shall be whirled at a rate of 1,400 to 1,500 r. p. m. or equivalent for 10 minutes. The volume of sediment at the bottom of each tube shall be read and recorded, estimating to 0.05 c. c. if possible. The tubes shall then be placed in the centrifuge, again whirled for 10 minutes as before, and removed for reading the volume of the sediment as before. This operation shall be repeated until the volume of sediment in each tube remains constant for three consecutive readings. In general, not more than four whirlings are required.

8. The volume of the solid sediment at the bottom of each centrifuge tube shall be read, estimating to 0.1 c. c. or closer, if possible. If the two readings differ by not more than 0.1 c. c., the mean of the two shall be reported as the "A. S. T. M. precipitation number." If the two readings differ by more than 0.1 c. c., two more determinations shall be made and the average of the four determinations shall be reported.

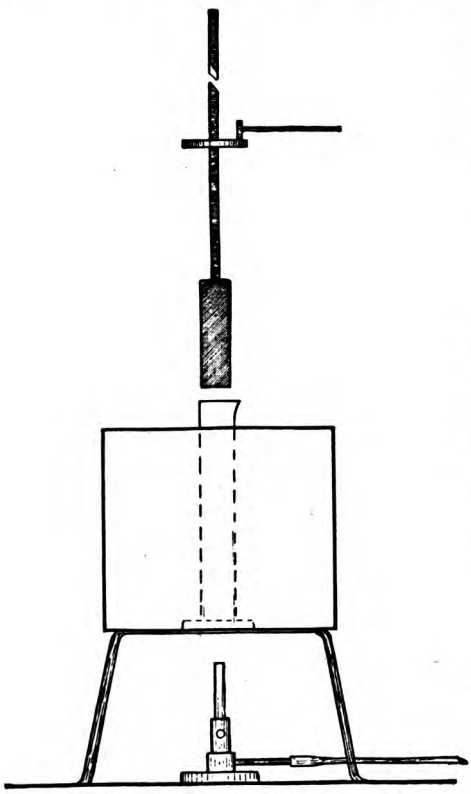

FIGURE 18.—Essential features of emulsifier.

ACCURACY.

9. With care and proper attention to details, duplicate determinations of precipitation number by this method should not differ by more than 0.1, provided the centrifuge tubes are accurate and readable to this degree.

METHOD 320.1.— EMULSION TEST AT 130° F.

APPARATUS.

1. A 100 c. c. graduated cylinder, $1\frac{1}{16}$ to $1\frac{1}{8}$ inches inside diameter.

2. An oil or water bath for maintaining the contents of the cylinder at the specified temperature.

3. A paddle consisting of a copper plate $4\frac{3}{4}$ inches long, between $\frac{3}{4}$ and $\frac{7}{8}$ inch wide, and $\frac{1}{16}$ inch thick.

4. Means for revolving this paddle about a vertical axis parallel to and midway between its two longer edges and for keeping the speed fairly constant at 1,500 r. p. m.

5. A stop so arranged that when the paddle is lowered into the cylinder the distance from the bottom of the paddle to the bottom of the cylinder will be about $\frac{1}{4}$ inch.

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PROCEDURE.

6. Place 40 c. c. of the emulsifying liquid in the 100 c. c. graduated cylinder and add 40 c. c. of the oil to be tested.

7. Heat the cylinder by means of the bath to 130° F.

8. Stir by rotating the paddle for five minutes at a speed of 1,500 r. p. m., stop the paddle, withdraw from the cylinder and wipe clean, returning to the cylinder as much of the emulsion as possible.

9. Allow the cylinder to stand at 130° F. for the specified time and then inspect. No continuous layer of emulsion shall remain.

METHOD 320.2.—EMULSION TEST AT 180° F.

APPARATUS AND PROCEDURE.

The apparatus and procedure are the same as in method 320.1, except that paragraphs 7 and 9 shall read:

7. Heat the cylinder by means of the bath to 180° F.

9. Allow the cylinder to stand at 180° F. for the specified time and then inspect. No continuous layer of emulsion shall remain.

METHOD 320.3.—DEMULSIBILITY AT 130° F.

APPARATUS.

Same as in test 320.1.

PROCEDURE.

6. Place 27 c. c. of the oil to be tested and 53 c. c. of distilled water in the cylinder.

7. Heat the cylinder by means of the bath to 130° F.

8. Stir by rotating the paddle for five minutes at a speed of 1,500 r. p. m., stop the paddle, withdraw from the cylinder, and wipe clean, returning to the cylinder as much of the emulsion as possible.

9. Keep the temperature of the cylinder constant at 130° F.

10. Take readings every minute of the position of the line of demarcation between the topmost layer of oil and the adjoining emulsion. Take the first reading one minute after stopping the paddle. With oils which act normally the rate of settling out of the oil increases up to a maximum and then decreases and the maximum value in cubic centimeters per hour is called the "demulsibility" and is recorded as the numerical result of the test. Each rate of settling is the average rate calculated from the time of stopping the paddle to the time of reading, as shown in the following condensed table:

Method of computing rate of settling.

Time.	Time since stopping paddle.	Reading at interface between oil and emulsion.	Oil settled out.	Rate of settling per hour.
	Minutes.		*C. c.*	*C. c.*
9.50	0	80	0	0
9.55	5	77	3	36
10.02	12	67	13	65
10.05	15	63	17	68
10.10	20	61	19	57

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The demulsibility in this case would be 68, the highest value in the last column. When the maximum rate of settling has not been reached at the end of one hour, the test is discontinued and the demulsibility taken as the number of cubic centimeters that settled out in the hour.

METHOD 320.4.—DEMULSIBILITY AT 180° F.

APPARATUS AND PROCEDURE.

The apparatus and procedure are the same as in test 320.3, except that paragraphs 7 and 9 shall read:

7. Heat the cylinder by means of the bath to 180° F.
9. Keep the temperature of the cylinder constant at 180° F.

METHOD 400.1.—PROTECTION TEST.

Coat a clean polished steel plate 2 inches long, $\frac{1}{2}$ inch wide, and $\frac{1}{8}$ inch thick, by immersing it in the sample to be tested, which has been heated to a temperature of 212° F. Remove the plate while it is still hot, allow to cool in a vertical position, and suspend vertically within a 10 per cent salt solution at room temperature for 30 days. At the end of this time rinse with gasoline and examine for corrosion.

METHOD 400.2.—OVEN SALT-SPRAY RAIN TEST.

Four bright steel plates, approximately 4 by 6 inches, shall be tacked to separate boards and thoroughly cleaned with benzene. The slushing oil to be examined shall be melted, if not already liquid, and an excess flowed over the plates. One of the plates shall be kept in a vertical position in the laboratory for 24 hours and then placed in a rack on the roof so that the plate shall be inclined at an angle of 45° to the vertical, facing south. The plate shall be sprayed lightly with a 3 per cent salt solution on the first day. The other three plates shall be hung in a vertical position in an oven and maintained at a temperature of 45° to 50° C. (113° to 122° F.) for at least 48 hours. The plates shall be removed from the oven, allowed to cool, and one plate exposed on the roof in the rack described above. Another plate shall be kept in a horizontal position and lightly sprayed with a 3 per cent salt solution once every day for a period of five days. The fourth plate shall be placed under an intermittent shower, a vigorous shower being applied for approximately three to four minutes, then no water for about the same length of time. The shower is to be formed by allowing water to siphon at intervals from a 5-gallon tank into a metal trough, the bottom of which has three or four parallel rows of small holes (about one-sixteenth inch in diameter). The water should fall about 2 feet from the trough to the plate. This test should be continued for not less than five hours, the plate being held in a position about 60° to the vertical immediately under the falling water. At the end of five hours of this intermittent showering the plate shall be placed in a horizontal position and allowed to remain with any adhering water for at least 24 hours. No rust shall be in evidence on any of the four plates after five days. When time permits, the plates on

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the roof should be exposed for a period of 60 days, and at the end of this time there should be no appreciable rust in evidence.

METHOD 410.1—BREAKDOWN TEST.

[A. S. T. M. Methods D48–17T and D117–21T.]

APPARATUS.

(*a*) Any well-designed high-tension testing transformer connected to an alternating current supply having as nearly a true sine wave as possible may be used. The transformer and the source of supply of energy shall not be less than 5 kv. a. The frequency shall not exceed 100 cycles per second.

(*b*) Regulation shall be so controlled that the high-tension testing voltage taken from the secondary of the testing transformer can be raised gradually from any point. The control may be made by generator field regulation, by the potentiometer method, with an induction regulator, or with a variable ratio auto transformer. Any method of regulating the voltage which does not distort the wave more than 10 per cent from a sinusoidal shape is satisfactory.

(*c*) The voltage may be measured by any approved method, preferably by connecting a voltmeter to an auxiliary ratio transformer coil, reading square root of mean square volts in the primary of the testing transformer. A volt-meter on the low-tension side of the transformer is satisfactory, if the ratio of transformation does not change any test condition. An electrostatic voltmeter properly calibrated in the high-tension circuit is also satisfactory.

(*d*) The sample shall be tested between polished brass or copper circular disk electrodes 1 inch in diameter, with a square edge. The electrodes shall be placed with their axes horizontal and coincident, and with a gap of 0.1 inch between their adjacent faces.

PROCEDURE.

(*a*) The electrodes and the containing vessel shall be wiped clean and thoroughly rinsed with oil-free, dry gasoline until they are entirely free from fibers. They shall then be rinsed at least once with a sample of the oil to be tested. The temperature of the gap and of the sample shall be between 20° and 30° C. (68° to 86° F.). The gap shall be filled with oil to a height of not less than 20 mm. (0.787 inch) above the top of the disks. Voltage shall be applied and increased uniformly at a rate of approximately 3,000 volts (rms.) per second until breakdown occurs as indicated by a continuous discharge across the gap. (Occasional momentary discharges which do not result in a permanent arc may occur; these should be disregarded.)

(*b*) Provision shall be made for opening the primary circuit as promptly as possible after breakdown has occurred in order to prevent unnecessary carbonization of the oil. After each puncture the testing vessel shall be jarred to loosen particles of carbon adhering to the electrodes and the oil gently agitated, but not with sufficient violence to introduce air bubbles.

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(*c*) Five breakdowns shall be made on each filling, after which the vessel shall be emptied and refilled with fresh oil from the original sample. The test shall be continued until the mean of the averages of at least three fillings is consistent within 10 per cent.

REPORT.

The report shall include the following:

(a) The volts (rms. value) at each "shot," the average voltage for each of the three or more fillings, and the grand average;

(*b*) The approximate temperature of the oil at the time of the test.

NOTE.—A precision of about 3 per cent may be reasonably expected in 15 tests, distributed among three consistent fillings taken in succession. But if the length of the gap is readjusted and possibilities of contamination exist, the precision may be only 6 or 7 per cent. Differences as great as 10 or 12 per cent may occur between different laboratories even where the work is carefully done.

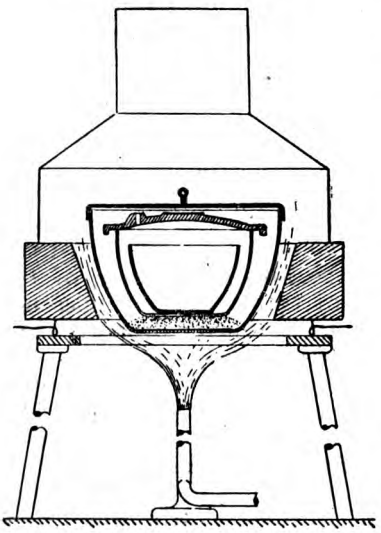

FIGURE 19.—Apparatus for determining carbon residue.

METHOD 500.1.—CARBON RESIDUE.

[A. S. T. M. Method D47–21.]

APPARATUS.

(*a*) Porcelain crucible, wide form, glazed throughout, 25 to 26 c. c. capacity, 46 mm. diameter.[8]

(*b*) Skidmore iron crucible, 45 c. c. (1½ ounces) capacity, 65 mm. in diameter, 37 to 39 mm. high with cover, without delivery tubes, and one opening closed.

(*c*) Wrought-iron crucible with cover, about 180 c. c. capacity, 80 mm. diameter, 58 to 60 mm. high. At the bottom of this crucible a layer of sand is placed about 10 mm. deep, or enough to bring the Skidmore crucible with cover on nearly to the top of the wrought-iron crucible.

(*d*) Triangle, pipe stem covered, projection on side to allow flame to reach the crucible on all sides.

(*e*) Sheet-iron or asbestos hood provided with a chimney about 2 to 2½ inches high, 2⅛ inches in diameter, to distribute the heat uniformly during the process.

(*f*) Asbestos or hollow sheet-iron block, 6 to 7 inches square, 1¼ to 1½ inches high, provided with opening in center 3¼ inches in diameter at the bottom and 3½ inches in diameter at the top.

METHOD OF TESTING.

The tests shall be conducted as follows: Ten grams of the oil to be tested are weighed in the porcelain crucible *a*, which is placed

[8] A glazed silica crucible of the same dimensions may be used.

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in the Skidmore crucible *b*. These two crucibles are set in the larger iron crucible *c*, being careful to have the Skidmore crucible set in the center of the iron crucible, covers being applied to the Skidmore and iron crucibles. Place on triangle and suitable stand with asbestos block and cover with sheet-iron or asbestos hood in order to distribute the heat uniformly during the process.

Heat from a Bunsen burner or other burner is applied with a high flame surrounding the large crucible *c* until vapors from the oil begin to ignite over the crucible, when the heat is slowed down so that the vapor (flame) will come off at a uniform rate. The flame from the ignited vapors should not extend over 2 inches above the sheet-iron hood. After the vapor ceases to come off, the heat is increased as at the start and kept constant for five minutes, making the lower part of large crucible red hot, after which the apparatus is allowed to cool somewhat before the crucible is uncovered. The porcelain crucible is removed, cooled in a desiccator, and weighed.

The entire process should require one-half hour to complete when heat is properly regulated. The time will depend somewhat upon the kind of oil tested, as a very thin, rather low flash-point oil will not take as long as a heavy, thick, high flash-point oil.

METHOD 510.1.—REACTION TEST.

Place 50 c. c. of the sample and 15 c. c. of distilled water in a 150-c. c. flask. Warm to 150° F. and shake thoroughly. Allow the mixture to cool and transfer 5 c. c. of the aqueous layer to each of two test tubes by means of a pipette. Add 1 drop of 1 per cent solution of methyl orange to the contents of one tube, and 1 drop of a 1 per cent solution of phenolphthalein to the other. No red or pink color shall result in either case.

METHOD 510.2.—ACIDITY IN GASOLINE.

NOTE.—This test is to be made immediately after distillation test 100.1.

Collect in a test tube the cooled residue from the distillation flask, add 3 volumes of distilled water, and shake the tube thoroughly. Allow the mixture to separate and remove the aqueous layer to a clean test tube by means of a pipette. Add 1 drop of a 1 per cent solution of methyl orange. No pink or red color shall be formed.

METHOD 510.3.—FREE ACID.

[A. S. T. M. Method D47–21.]

METHOD OF TESTING.

(*a*) *For fatty oils.*—Accurately weigh 10 grams of the oil into a flask, add 50 c. c. of 95 per cent alcohol, which has been neutralized with weak caustic soda, and heat to the boiling point. Agitate the flask thoroughly in order to dissolve the free fatty acids as completely as possible. Titrate while hot with aqueous tenth-normal

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alkali, free from carbonate, using phenolphthalein, alkali blue, or turmeric as an indicator. Agitate thoroughly after each addition of alkali.

To express results as percentage of oleic acid, use the following equation:

1 c. c. of tenth-normal alkali=0.0282 gram of oleic acid.

Alkali, 1 c. c. of which is equivalent to 0.5 per cent of oleic acid, may be used.

(*b*) *For mineral oils.*—Follow the same method for mineral oils except for the alcohol, which should consist of equal proportions of distilled water and 95 per cent alcohol.

In titrating dark-colored mineral oils, the end reaction is better observed when alkali blue 6 B. is used as the indicator.

METHOD 520.1.—SULPHUR IN BURNING OILS.

[A. S. T. M. Method D90–21T.]

APPARATUS.

Absorber of chemically resistant glass, about 150 c. c. capacity, containing glass beads or short pieces of glass rod in the suction side as shown.

Chimney of chemically resistant glass connected with the absorber by a rubber stopper.

Spray trap of chemically resistant glass connected with the absorber by a rubber stopper.

Small lamp of about 25 c. c. capacity. This lamp may conveniently consist of a 25 to 35 c. c. Erlenmeyer flask and a cork carrying a short section of glass tubing about $\frac{1}{8}$ inch in inside diameter. The cork must be grooved along the sides so that air may enter the flask while the oil is being consumed.

Ordinary cotton wicking.

Filter pump or other means for continuous suction and rubber tubing to connect with spray trap.

SOLUTIONS REQUIRED.

Hydrochloric acid.—Solution containing 2.275 grams HCl per liter, carefully checked for accuracy.

Sodium carbonate.—Solution containing 3.306 grams Na_2CO_3 per liter. Exactly 10.0 c. c. should be required to neutralize 10.0 c. c. of the hydrochloric acid solution.

Methyl orange.—Solution in distilled water, containing 0.004 gram methyl orange per liter.

PROCEDURE.

Pass two strands of new cotton wicking about 4.5 inches long through the $\frac{1}{8}$-inch diameter wick tube so that they are not twisted but parallel in the wick tube. Trim the wick with very sharp scissors. Pour into the clean, dry lamp about 20 c. c. of the oil to be tested, insert the wick, and cork and weigh the assembly with

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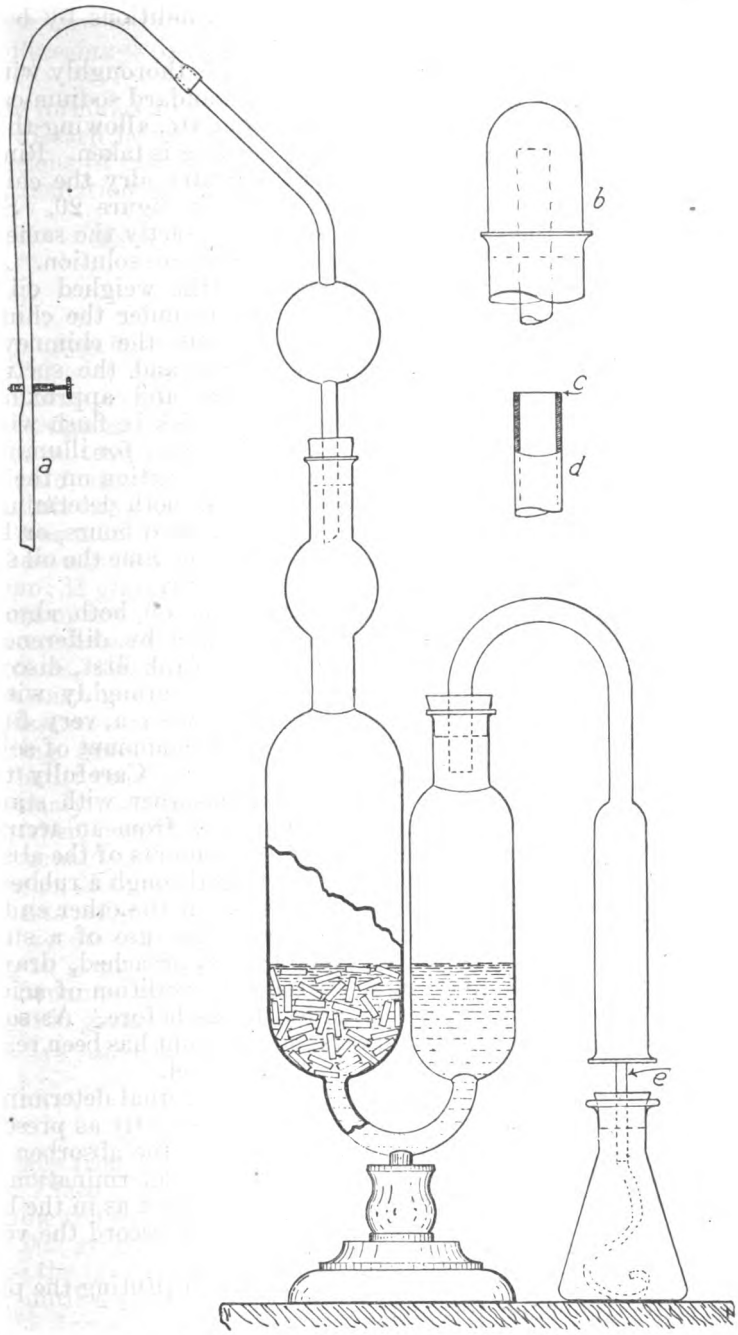

FIGURE 20.—Apparatus for determination of sulphur in oils (A. S. T. M.): *a*, Suction tube; *b*, top of burner with glass cap in place; *c*, annealed top; *d*, part section of glass burner; *e*, glass burner.

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an accuracy of 0.001 gram. It is advisable to make a blank determination at the same time and under the same conditions by burning sulphur-free alcohol in a similar lamp.

Rinse the absorber containing the glass beads thoroughly with distilled water and add exactly 10.0 c. c. of the standard sodium-carbonate solution from an accurately calibrated burette, allowing the burette to drain for three minutes before the reading is taken. Rinse the chimney and the spray trap with distilled water, dry the chimney, and connect both to the absorber as shown in figure 20. Set up the apparatus for the blank determination in exactly the same manner, using exactly 10.0 c. c. of the sodium-carbonate solution. Apply gentle suction to both absorbers, light both the weighed oil lamp and alcohol lamp, and then place in position under the chimneys, so that the tops of the wick tubes extend into the chimneys not more than $\frac{1}{16}$ inch. Adjust the wick height and the suction so that the flame is steady, free from smoke, and approximately one-fourth inch high. This requires that the wick be flush with the top of the wick tube for naphthas and a little higher for illuminating oils. The room must be free from drafts. The suction on the blank should be so adjusted that air is drawn through both determinations at the same rate. Continue burning for about two hours, or less if the sulphur content of the oil is high. During this time the oil should be consumed at the rate of about 1 gram per hour.

Extinguish the flames and stop the suction on both absorbers. Weigh the oil lamp immediately and calculate by difference the weight of oil consumed. Working with the blank first, disconnect the spray trap and chimney and wash them thoroughly with the methyl orange solution, using a wash bottle with a very fine jet and collecting the washings in the absorber. The amount of solution required for washing should not exceed 35 c. c. Carefully titrate the very faintly yellowish solution in the absorber with standard HCl, added to the suction side of the absorber from an accurately calibrated burette. During this titration the contents of the absorber should be agitated carefully, either by blowing through a rubber tube held between the operator's lips and connected at the other end with the chimney side of the absorber or else by the use of a suitable rubber syringe bulb. As the end point is approached, draw the liquid back into the chimney side between each addition of acid and then blow it into the suction side, agitating as before. As soon as the first permanent pink color appears the end point has been reached. Read and record the volume of HCl solution used.

Rinse the chimney and spray trap used in the actual determination into the absorber to which they were connected, exactly as prescribed for the blank. If the methyl orange solution in the absorber has a pink color, too much oil has been burned and the determination must be repeated, burning for a shorter time. Titrate just as in the blank, making sure that the absorber is cold. Read and record the volume of HCl solution required.

Calculate the sulphur content of the oil by substituting the proper values in the following formula:

$$\text{Percentage of sulphur} = \frac{(\text{HCl for blank, c.c.} - \text{HCl for sample, c.c.}) \times 0.1}{\text{grams of oil burned.}}$$

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If blank is not run, the formula is:

$$\text{Percentage of sulphur} = \frac{(Na_2CO_3, \text{ c. c.} - HCl, \text{ c. c.}) \times 0.1}{\text{grams of oil burned.}}$$

These formulas are correct only for the standard solutions specified, 1 c. c. of each being equivalent to 0.001 gram of sulphur. The use of solutions of other strength, such as N/10, involves more complicated calculation and is not advisable.

METHOD 520.2.—SULPHUR IN FUEL OIL.

APPARATUS.

1. An oxygen bomb satisfying the following requirements:
(*a*) Capacity not less than 300 c. c.
(*b*) Entire interior surface chemically resistant.
(*c*) Design and construction such that no leaks shall occur at any pressure or temperature generated during use, and such that when open liquid contents can be easily and completely drained.

2. An oil cup of platinum or glazed silica with a capacity not less than 2.5 or more than 5 c. c.

3. *Fuse wire.*—If platinum oil cup is used, fuse wire must be of platinum; if glazed silica oil cup is used, fuse wire may be of either platinum or iron. No. 35 B. & S. gage is a convenient size.

4. Accessories for bomb, such as oxygen supply, gage, charging connections, bench plate, wrench, bucket of water, and electrical connections.

5. *Glassware and supplies.*—Beakers from 300 to 500 c. c. capacity; measuring graduates or pipettes; filtering funnels; hot plate or steam bath; crucibles and furnace suitable for ignition of precipitates; analytical balance sensitive to 0.0002 gram; "qualitative" filter paper, preferably 11-cm. size; "ashless quantitative" filter paper, capable of holding barium sulphate, preferably 9-cm. size (Whatman No. 42, S. and S., No. 589 Blue Ribbon, and A. D. Little are brands known to be satisfactory.)

6. *Reagents.*—Distilled water. Sodium carbonate solution, containing 50 grams Na_2CO_3 per liter. Hydrochloric acid, c. p., sp. gr. 1.20. Bromine water (saturated). Barium chloride solution containing 100 grams of $BaCl_2.2H_2O$ per liter.

NOTE.—All reagents must be sulphur free.

PROCEDURE.

7. Place from 10 to 20 c. c. of distilled water in the bottom of the bomb. Place in the oil cup 0.6 to 0.8 gram of the oil to be tested and determine the weight of this charge to an accuracy of at least plus or minus .002 gram. Place the cup in the proper position in the bomb, arrange the ignition mechanism, and close the bomb. Admit oxygen slowly until a pressure of 40 atmospheres is reached. Attach leads from the firing circuit, place the bomb in a bucket of cold water, and ignite. Allow to stand 10 minutes, and then remove from the water. Open the valve of the bomb and allow the gas to escape at an approximately even rate, so that the pressure is reduced to atmospheric in

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not less than one minute. Open the bomb and rinse all parts of its interior, including the oil cup, with a fine jet of distilled water. Collect all washings, which should not amount to more than 350 c. c., in a beaker. *Particular care must be taken not to lose, by splashing or otherwise, any of the liquid contents of the bomb.* Add 10 c. c. of the sodium-carbonate solution to the washings, heat almost to boiling, and keep at this temperature for five minutes. Filter through a washed "qualitative" filter paper. Wash the filter thoroughly. Add 2 c. c. of the concentrated HCl and 10 c. c. of saturated bromine water to the filtrate. Evaporate to about 75 c. c. on the steam bath or hot plate. To the hot solution add in a fine stream or dropwise 10 c. c. of hot barium-chloride solution. Stir during the addition and for two minutes afterwards. Allow to stand overnight, or keep hot for one hour on the steam bath or hot plate, allowing the precipitate to settle for an hour while cooling. Filter the supernatent liquid through the "ashless quantitative" filter paper, wash the precipitate with hot water, first by decantation, then on the filter, till free from chloride. Transfer the paper and precipitate to a suitable crucible, dry at low heat until moisture is evaporated, char the paper (without flaming), and finally ignite at a good red heat until the precipitate is just burned white. (A satisfactory means of accomplishing these operations is to place the crucible containing the wet filter paper in a cold electric muffle furnace and turn on the current. Drying, charring, and ignition will usually occur at the desired rate.)

After ignition is complete allow the crucible to cool to room temperature and weigh. The use of a dessicator is not recommended.

From the increase in weight of the crucible calculate the percentage of sulphur as follows:

$$\text{Percentage of sulphur} = \frac{\text{grams of } BaSO_4 \times 13.734}{\text{grams of oil used.}}$$

METHOD 520.3.—DOCTOR TEST (SODIUM PLUMBITE).

PREPARATION OF REAGENTS.

(1) *Sodium plumbite* (*doctor solution*).—Dissolve approximately 125 grams of sodium hydroxide (NaOH) in a liter of distilled water. Add 60 grams of litharge (PbO) and shake vigorously for 15 minutes or let stand with occasional shaking for at least a day. Allow to settle and decant or siphon off the clear liquid. Filtration through a mat of asbestos may be employed if the solution does not settle clear. The solution should be kept in a tightly corked bottle and should be refiltered before use if not perfectly clear.

(2) *Sulphur.*—Pure, dry flowers of sulphur.

MAKING OF TEST.

Shake vigorously together in a test tube 10 c. c. of the sample to be tested and 5 c. c. of sodium plumbite solution for about 15 seconds. Add a small pinch of flowers of sulphur, again shake for 15 seconds, and allow to settle. The quantity of sulphur used should be such that practically all of it floats on the interface between the sample and the sodium plumbite solution.

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INTERPRETATION OF RESULTS.

If the sample is discolored, or if the yellow color of the sulphur film is noticeably masked, the test shall be reported as *positive* and the sample condemned as "sour." If the sample remains unchanged in color, and if the sulphur film is bright yellow or only slightly discolored with gray or flecked with black, the test shall be reported *negative* and the sample considered "sweet."

METHOD 530.1.—CORROSION TEST (COPPER DISH).

APPARATUS.

A freshly polished hemispherical dish of spun copper approximately $3\frac{1}{2}$ inches in diameter.

MAKING OF TEST.

Place 100 c. c. of the gasoline to be examined in the dish and place the dish in an opening of an actively boiling steam bath, so that the steam comes in contact with the outer surface of the dish up to the level of the gasoline. Leave the dish on the steam bath until all volatile material has disappeared.

INTERPRETATION OF RESULTS.

If the gasoline contains dissolved elementary sulphur or corrosive sulphur compounds, the bottom of the dish will be colored gray or black.

If the gasoline contains undesirable gum-forming constituents, there will be a weighable amount of gum deposited on the dish. Acid residues will show as gum in this test.

METHOD 530.2.—CORROSION TEST AT 122° F.

[Copper Strip.]

Place a clean strip of mechanically polished pure sheet copper, about one-half inch wide and 3 inches long and 10 c. c. of the sample, in a clean test tube. Close the tube with a vented stopper and hold in a thermostat for three hours at 122° F. Rinse the copper strip with sulphur-free acetone and compare it with a similar strip of freshly polished copper. Discoloration or pitting indicates corrosion.

METHOD 530.3.—CORROSION TEST AT 210° F.

[Copper Strip.]

This is the same as method 530.2 except that the tube is held at 212° F. instead of at 122° F.

METHOD 530.4.—FIVE-DAY CORROSION TEST.

Place clean strips of mechanically polished sheet brass, sheet steel, and sheet aluminum, about $\frac{1}{2}$ inch wide and 3 inches long, in clean test tubes and add 10 c. c. of the sample to be tested to each tube. Close the tubes with vented stoppers and hold in a thermostat for

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five days at 210° F. At the end of this time rinse each strip with sulphur-free acetone and compare it with a similar strip of the same metal freshly polished. Discoloration or pitting indicates corrosion.

METHOD 540.1.—SAPONIFICATION NUMBER.

[A. S. T. M. Method D94–21T.]

SOLUTIONS REQUIRED.

Alcoholic solution for saponification.—Dissolve 58 grams of potassium hydrate "purified by alcohol" in 500 c. c. of 95 per cent purified ethyl alcohol. Allow the solution to settle in a dark place. Draw off the clear solution or filter through an asbestos filter and make up to 1 liter with 95 per cent alcohol. The solution so prepared shall stand at least 20 hours before it is standardized.

Alcohol.—Purify 95 per cent ethyl alcohol with silver oxide in the following manner:

Dissolve 1.5 grams of c. p. silver nitrate in about 3 c. c. of water, add to 1 liter of alcohol in a glass-stoppered bottle and mix thoroughly. Dissolve 3 grams of potassium hydrate (by alcohol) in 10 to 15 c. c. of warm alcohol. After cooling, add slowly to the alcoholic silver-nitrate solution, stirring slightly. Allow the precipitated silver oxide to settle, siphon off the clear solution, and distill on a steam bath.

Standard hydrochloric-acid solution.—One-half normal solution.

Phenolphthalein solution.—One gram phenolphthalein in 100 c. c. alcohol and water.

APPARATUS.

The saponification shall be carried out in a wide-mouthed flat-bottom extraction flask, or Erlenmeyer flask, of 250 to 300 c. c. capacity, fitted to a reliable condenser properly connected with a good cork. The boiling shall preferably be carried on by means of an electric hot plate.

BLANK DETERMINATION.

Determination shall be made in duplicate in the alcoholic-potash solution in the following manner:

Measure accurately into the flask 25 c. c. of alcoholic-potash solution from a calibrated pipette. The tip and outside of the pipette shall be wiped off with a clean filter paper before the solution is delivered, then rinsed out with 25 c. c. of neutral alcohol. If a standard burette is used, allow 60 seconds total time for drawing and draining. Connect the flask to a suitable condenser and boil for three hours. Before disconnecting the flask wash out the condenser with a few cubic centimeters of neutral alcohol; if a Soxhlet is used as the condenser the tip shall be washed off into the flask. Titrate while hot with N/2 HCl, using three drops phenolphthalein indicator. The total number of cubic centimeters of N/2 HCl required for the blank represents the strength of the alcoholic-potash solution.

PROCEDURE.

For straight fats or oils use 2 or 3 grams of the material, for oils containing over 30 per cent of fatty oils use about 5 grams, and for oils containing less than 30 per cent of fatty oils use about 10 grams.

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Weight the oil accurately, by difference, from a small beaker into the saponification flask. Add 25 c. c. of alcoholic potash solution and 25 c. c. of neutral alcohol in the same manner as for the blank, connect to the condenser, and boil for three hours. Titrate while hot. Calculate the saponification number from the difference between the number of cubic centimeters of N/2 HCl required for the determination and the average of the two blanks, using the formula:

$$\text{Saponification number} = \frac{\text{difference, cubic centimeters} \times 28.05}{\text{weight of oil, grams}}$$

Petrolic ether may be used with compounded cylinder oils in the quantity of 50 c. c., providing a Soxhlet extraction flask is used to collect this petrolic ether periodically. The Soxhlet flask should be so adjusted, through the addition of glass rodding or beads when necessary, that it will just overflow with the full quantity of petrolic ether.

The percentage of fatty oil (or fat) in a compounded petroleum product can be calculated from the saponification number of such a product only when the saponification number of the fatty oil is known. If the saponification numbers of both fatty oil and compounded oil are known, the following formula should be used:

$$\text{Percentage of fatty oil} = \frac{100 \times \text{saponification number of compounded oil}}{\text{saponification number of fatty oil}}$$

For this determination the following values of saponification number may be used (see method 540.2):

Fatty oil.	Saponification number.	Fatty oil.	Saponification number.
Lard oil	192–198	Soya bean	189–197
Tallow	193–198	Peanut	186–197
Neat's-foot	193–204	Cottonseed	191–197
Fish	140–193	Blown rapeseed	195–216
Sperm	120–140	Blown cottonseed	210–225
Castor	176–187	Degras	110–210
Rapeseed	170–179		

METHOD 540.2.—FATTY OIL.

The percentage of fatty oil shall be calculated from the saponification number (method 540.1) by means of the following formula:

$$\text{Percentage of fatty oil} = \frac{100 \times \text{saponification number of the sample}}{\text{saponification number of fatty oil}}$$

The saponification number of the fatty oil used in compounding shall be assumed to be 195, unless the manufacturer of the sample under examination submits an affidavit stating the kind of fatty oil used and its saponification number.

METHOD 550.1.—UNSATURATION IN GASOLINE.

APPARATUS.

1. An unsaturation test bottle as shown in figure 21. The graduated portion of the neck shall contain 10 c. c. (± 0.05 c. c.) at a temperature of 20° C. and shall be graduated in 2 per cent divisions

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and numbered every 10 per cent from the bottom up, the first and each succeeding 10 per cent line to be longer than the others.

2. A pipette, calibrated to deliver 10 c. c. (±0.05 c. c.) of aviation gasoline at a temperature of 20° C.

3. A 20-c. c. graduate.

MAKING OF TEST.

With the pipette place 10 c. c. of the gasoline to be tested in a clean, dry unsaturation test bottle, cool for two minutes by immersing in ice water, then add 20 c. c. of commercial 66° sulphuric acid (containing approximately 93.2 per cent H_2SO_4) from the graduate. Care should be taken that the acid runs quietly down the side of the bottle instead of splashing onto the surface of the gasoline. Close the bottle with a rubber stopper and shake, first slowly, then vigorously with a rotary motion for five minutes. Separate the gasoline and the acid by either of the following methods:

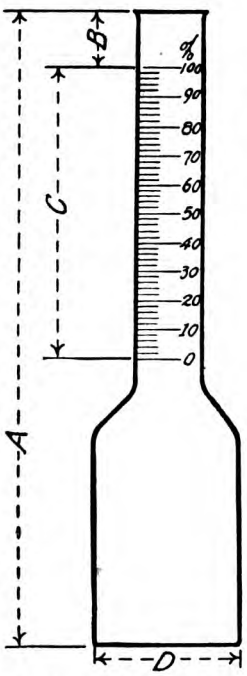

FIGURE 21. — Modified Babcock bottle for unsaturation test of gasoline. Approximate dimensions: *A*, 6½ inches; *B*, ½ inch; *C*, 2½ to 3 inches; *D*, 1 1/16 inches.

Gravity separation.—Add sulphuric acid to the contents of the bottle until the surface of the liquid is level with the upper graduation mark on the neck. Stopper tightly and allow the bottle and contents to stand 12 hours. Report the percentage loss of volume of the gasoline as percentage of unsaturation.

Centrifugal separation.—Place the stoppered bottle in a suitable centrifuge and whirl it for two or three minutes at a speed of 500 to 1,000 revolutions per minute. Add sulphuric acid until the surface of the liquid is level with the lower graduation mark and again centrifuge. Add acid to bring the liquid to the upper graduation mark, and report the unsaturation as above.

METHOD 550.2.— UNSATURATION IN TRANSFORMER OIL.

APPARATUS.

(1) A 25 c. c. glass stoppered cylinder, graduated to 0.2 c. c.

(2) A graduated burette.

MAKING OF TEST.

Measure out 15 c. c. of the sample in the glass cylinder, cool in ice water for 5 minutes, add from the burette 10 c. c. of c. p. sulphuric acid of 1.84 specific gravity, and again cool in ice water for 10 minutes. Tightly stopper the cylinder and shake violently for 15 seconds; again place in ice water until thoroughly cool and shake violently for one minute. After this has stood five minutes remove the stopper, place the cylinder in a suitable centrifuge, and whirl until the oil becomes clear and no longer increases in volume. Allow to

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stand until it comes to room temperature and note the volume of the oil absorbed by the sulphuric acid. Calculate the per cent for the 15 c. c. of original oil, and report as percentage of unsaturation.

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